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1296811

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*March 16, 2005*

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**APPLICATION NUMBER:** *60/525,741*
**FILING DATE:** *December 01, 2003*
**RELATED PCT APPLICATION NUMBER:** *PCT/US04/40074*

Certified by

Jon W. Dudas

Under Secretary of Commerce
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PTO/SB/16 (10-01)
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# PROVISIONAL APPLICATION FOR PATENT COVER SHEET

**This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).**

**Express Mail Label N .**

| INVENTOR(S) | | |
|---|---|---|
| Given Name (first and middle [if any]) | Family Name or Surname | Residence (City and either State or Foreign Country) |
| Tompa, Gary S.<br>Rice, Catherine E.<br>Sbrockey, Nick M.<br>Provost, Lloyd G. | | Bell Mead, NJ<br>Scotch Plains, NJ<br>Gaithersburg, MD<br>GlenRidge, NJ |

☐ *Additional inventors are being named on the _____ separately numbered sheets attached hereto*

1754 U.S.PTO 60525741 120103

**TITLE OF THE INVENTION (500 characters max)**

Passive and Active Optical Film Structure(s) Making

*Direct all correspondence to:* **CORRESPONDENCE ADDRESS**

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OR

| | | | | | |
|---|---|---|---|---|---|
| ☐ Firm or Individual Name | Structured Materials Industries, Inc. | | | | |
| Address | 201 Circle Drive North, Unit 102/103 | | | | |
| Address | | | | | |
| City | Piscataway | State | NJ | ZIP | 08854 |
| Country | U.S | Telephone | 732-302-9274 | Fax | 732-302-9275 |

**ENCLOSED APPLICATION PARTS *(check all that apply)***

☑ Specification *Number of Pages* _____ ☐ CD(s), Number _____

☐ Drawing(s) *Number of Sheets* _____ ☐ Other (specify) _____

☐ Application Data Sheet. See 37 CFR 1.76

**METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT**

☑ Applicant claims small entity status. See 37 CFR 1.27.

☑ A check or money order is enclosed to cover the filing fees

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FILING FEE AMOUNT ($)

The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

☐ No.

☐ Yes, the name of the U.S. Government agency and the Government contract number are: _____

*Respectfully submitted,*

SIGNATURE 11/24/03

Date 11/24/2003

TYPED or PRINTED NAME Dr. Gary S. Tompa

REGISTRATION NO. *(if appropriate)*

Docket Number:

TELEPHONE 732-302-9274

## USE ONLY FOR FILING A PROVISIONAL APPLICATION FOR PATENT

This collection of information is required by 37 CFR 1.51. The information is used by the public to file (and by the PTO to process) a provisional application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 8 hours to complete, including gathering, preparing, and submitting the complete provisional application to the PTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, Washington, D.C. 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Box Provisional Application, Assistant Commissioner for Patents, Washington, D.C. 20231.

# *PROVISIONAL APPLICATION COVER SHEET*
## *Additional Page*

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# FEE TRANSMITTAL for FY 2003

*Effective 01/01/2003. Patent fees are subject to annual revision.*

☑ Applicant claims small entity status. See 37 CFR 1.27

**TOTAL AMOUNT OF PAYMENT** ($)

| *Complete if Known* | |
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| Application Number | |
| Filing Date | |
| First Named Inventor | |
| Examiner Name | |
| Art Unit | |
| Attorney Docket No. | |

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☐ Charge fee(s) indicated below, **except for the filing fee** to the above-identified deposit account.

## FEE CALCULATION

### 1. BASIC FILING FEE

| Large Entity Fee Code | Large Entity Fee ($) | Small Entity Fee Code | Small Entity Fee ($) | Fee Description | Fee Paid |
|---|---|---|---|---|---|
| 1001 | 750 | 2001 | 375 | Utility filing fee | |
| 1002 | 330 | 2002 | 165 | Design filing fee | |
| 1003 | 520 | 2003 | 260 | Plant filing fee | |
| 1004 | 750 | 2004 | 375 | Reissue filing fee | |
| 1005 | 160 | 2005 | 80 | Provisional filing fee | 80 |

SUBTOTAL (1) ($) 80

### 2. EXTRA CLAIM FEES FOR UTILITY AND REISSUE

| | | Extra Claims | | Fee from below | Fee Paid |
|---|---|---|---|---|---|
| Total Claims | | -20** = | | X | = |
| Independent Claims | | - 3** = | | X | = |
| Multiple Dependent | | | | | = |

| Large Entity Fee Code | Large Entity Fee ($) | Small Entity Fee Code | Small Entity Fee ($) | Fee Description |
|---|---|---|---|---|
| 1202 | 18 | 2202 | 9 | Claims in excess of 20 |
| 1201 | 84 | 2201 | 42 | Independent claims in excess of 3 |
| 1203 | 280 | 2203 | 140 | Multiple dependent claim, if not paid |
| 1204 | 84 | 2204 | 42 | ** Reissue independent claims over original patent |
| 1205 | 18 | 2205 | 9 | ** Reissue claims in excess of 20 and over original patent |

SUBTOTAL (2) ($) 0

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## FEE CALCULATION (continued)

### 3. ADDITIONAL FEES

| Large Entity Fee Code | Large Entity Fee ($) | Small Entity Fee Code | Small Entity Fee ($) | Fee Description | Fee Paid |
|---|---|---|---|---|---|
| 1051 | 130 | 2051 | 65 | Surcharge - late filing fee or oath | |
| 1052 | 50 | 2052 | 25 | Surcharge - late provisional filing fee or cover sheet | |
| 1053 | 130 | 1053 | 130 | Non-English specification | |
| 1812 | 2,520 | 1812 | 2,520 | For filing a request for *ex parte* reexamination | |
| 1804 | 920* | 1804 | 920* | Requesting publication of SIR prior to Examiner action | |
| 1805 | 1,840* | 1805 | 1,840* | Requesting publication of SIR after Examiner action | |
| 1251 | 110 | 2251 | 55 | Extension for reply within first month | |
| 1252 | 410 | 2252 | 205 | Extension for reply within second month | |
| 1253 | 930 | 2253 | 465 | Extension for reply within third month | |
| 1254 | 1,450 | 2254 | 725 | Extension for reply within fourth month | |
| 1255 | 1,970 | 2255 | 985 | Extension for reply within fifth month | |
| 1401 | 320 | 2401 | 160 | Notice of Appeal | |
| 1402 | 320 | 2402 | 160 | Filing a brief in support of an appeal | |
| 1403 | 280 | 2403 | 140 | Request for oral hearing | |
| 1451 | 1,510 | 1451 | 1,510 | Petition to institute a public use proceeding | |
| 1452 | 110 | 2452 | 55 | Petition to revive - unavoidable | |
| 1453 | 1,300 | 2453 | 650 | Petition to revive - unintentional | |
| 1501 | 1,300 | 2501 | 650 | Utility issue fee (or reissue) | |
| 1502 | 470 | 2502 | 235 | Design issue fee | |
| 1503 | 630 | 2503 | 315 | Plant issue fee | |
| 1460 | 130 | 1460 | 130 | Petitions to the Commissioner | |
| 1807 | 50 | 1807 | 50 | Processing fee under 37 CFR 1.17(q) | |
| 1806 | 180 | 1806 | 180 | Submission of Information Disclosure Stmt | |
| 8021 | 40 | 8021 | 40 | Recording each patent assignment per property (times number of properties) | |
| 1809 | 750 | 2809 | 375 | Filing a submission after final rejection (37 CFR 1.129(a)) | |
| 1810 | 750 | 2810 | 375 | For each additional invention to be examined (37 CFR 1.129(b)) | |
| 1801 | 750 | 2801 | 375 | Request for Continued Examination (RCE) | |
| 1802 | 900 | 1802 | 900 | Request for expedited examination of a design application | |

Other fee (specify) _____

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## SUBMITTED BY

(Complete *(if applicable)*)

| | | | | | |
|---|---|---|---|---|---|
| Name (Print/Type) | Dr. Gary S. Tompa | Registration No. (Attorney/Agent) | | Telephone | 732-302-9274 |
| Signature | | | | Date | 11/24/03 |

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732 302 V9274 F9275

**BUILDING A BETTER WORLD THROUGH SCIENCE, UNDERSTANDING AND COMMERCIALIZATION**

November 24, 2003

Box Provisional Application
Assistant Commissioner for Patents
Washington, D.C. 20231

RE: Patent provisional

Dear Patent Officer,

Enclosed Please find our provisional patent application including:

1. Provisional Patent Applications SBIR proposals numbered 41413, 41415, 41469, 41474
2. All incremental and final reports for 41413 and 41415
3. Some supplemental viewgraphs
4. A set of draft claims
5. Other support documents
6. Payment

Sincerely,

Dr. Gary S. Tompa

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Passive and Active Optical Film Structure Making

Tompa, Rice, Skorchey, Provost

11/23/03 & all 4 proposals → 41468, 41415, 41469, 41474 & misc. figure system schematics.

Claims for $LiNbO_3$ MOCVD process

A system for depositing $LiNbO_3$ films, both amorphous and crystalline

~~and ... process~~

A system for controlling composition to give stoichiometric or congruent $LiNbO_3$ or compositions in between as desired.

– a system for controlling composition to a defined function

A system for controlling uniformity.

– temperature
– precursor flux

A process for depositing amorphous $LiNbO_3$.

A process for depositing crystalline $LiNbO_3$.

A process for depositing Li-containing complex oxides other than $LiNbO_3$.

A process for depositing thick (>1 micron) Li-containing oxides.

A process for depositing oriented crystalline $LiNbO_3$.

A process for depositing $LiNbO_3$ doped-as-grown with titanium or other metals to tailor the index or other properties.

A process for depositing $LiNbO_3$ with very low Fe impurities for resistance to optical damage due to the photorefractive effect.

A process for depositing an epitaxial crystalline film on crystalline substrates such as $LiNbO_3$, sapphire, and others with closest packed oxide layers that can serve as a template. Other potential epitaxial substrates include perovskites such as $LaAlO_3$ and $SrTiO_3$, ZnO, and many others.

A process for depositing $LiNbO_3$ as an amorphous layer on substrates such as $LiNbO_3$ and all others considered for epitaxy, as well as quartz, other silicates, ITO, silicon, basically any solid substrate that does not give rise to excessive interfacial reactions with the film.

A process for depositing $LiNbO_3$ films on metal substrates such as Pt or Ni or Cr.

A process for creating multilayer structures incorporating amorphous or crystalline $LiNbO_3$ layers along with dielectric layers (such as $CeO_2$ other oxides or non-oxides), conducting layers, or other layers crystalline or amorphous, with the $LiNbO_3$ layer being either underneath or on top of the other layers.

A method for depositing layers with controlled index profile through control of doping profiles. and/or composition.

these processes wherein selected precursors are dissolved in a solvent that is brought into a flash evaporator (or heated plenum) & converted to vapor that is transferred to the ~~reactor~~ reactor containing a heated substrate whereat a thin film grows (see schematic figure). One or several sources and variable rates with conventional or gas sources may be used to vary the film structure

dielectric overcoat / layered structures – multi-layered dielectrics

TCO overcoat / layered structures

NOB - Sequence of growing layers may be interspaced with annealing amorphous growth films to single crystals or patterning and etching amorphous films before (or after) annealing to a single crystal or poly

This relates to L. McClenahan's prior process patent work.

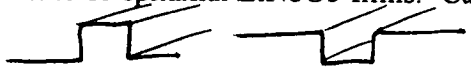
A method for depositing $LiNbO_3$ layers as cladding over existing optical device structures in $LiNbO_3$ bulk or in other optical materials.

as well as photonic crystals

Claims for MOCVD $LiNbO_3$ thin film devices

Active or Passive

Waveguide devices made in amorphous, polycrystalline or epitaxial LiNbO3 films. Can be ridge waveguides or channel waveguides.



Mach-Zehnder type interferometers made in amorphous, polycrystalline or epitaxial LiNbO3 films

doped or other materials entirely

Photonic crystal devices made in epitaxial LiNbO3 films,

c.+ multiple layers or combinations of multiple layers

a. using air as the confining medium
b. using doped LiNbO3 as the confining medium
c. using an entirely different material as the confining medium

The photonic devices made in LiNbO3 films that are used for:

d. waveguides
e. filters
f. detector
g. mixers
h. other photonic crystal

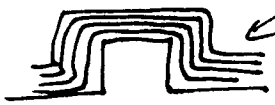
Pattern away

metal

TCO

metal

dielectric stack to reflect light into the waveguide



Light sources made by controlled doping of epitaxial LiNbO3 films with elements such as Er

Optical detectors made by controlled doping of epitaxial LiNbO3 films with elements such as Er

Opto-electronic integrated circuits (OEIC) using LiNbO3 films, either amorphous, polycrystalline or epitaxial, as optical interconnects

(functional)

Any optical or electro-optical device built by using a LiNbO3 thin film, with a change in composition, either abrupt or gradual, in which the composition change is used to produce a change in index of refraction, in order to confine a light wave.

Any electro-optical device built by using the property of a LiNbO3 thin film to change its optical properties under the influence of an applied electric field, in order to manipulate an optical signal.

Any electro-optical devices that use a LiNbO3 thin film, as a material with electrically controllable optical properties, in combination with a transparent conductive oxide layer, which is used to apply an electrical field to the LiNbO3. The LiNbO3 film and the transparent conductive oxide may or may not be deposited consecutively in the same deposition chamber.

copy

Any electro ---- a dielectric film or stack of films to focus light into the electro-optic device or the selected absence of such films to allow light out.

Background Info for Patent

Problem to be solved
- there is a need for devices to control fiber optical communications
  - such as optical switches, modulators
  - also devices for wavelength division multiplexing (WDM)
    - control multiple signals over same fiber
    - multiplexer, with add/drop capabilities

Prior art
- LiNbO3 is best material for electro-optical devices in many cases or near best in other cases
  - high transparency
  - high EO coefficient
  - non-linear optical properties
- There is presently no good process for making thin films of LNbO3
  - presently only bulk devices with diffused layers to define waveguides
    - result is poor confinement
    - large, slow, high voltage devices
- There is a need for a viable thin film technology for LiNbO3
  - Need for both the deposition tools and the process

- Previous attempts to make LiNbO3 thin films
  - films were too thin for practical devices
  - films crack and peeled from substrate
  - deposition rate was too slow.

SMI's approach
- develop hardware and process that can make practical LiNbO3 films
- SMI Hardware solution
  - MOCVD system designed specifically for depositing LiNbO3 thin films
    - Flash Evaporator
    - Rotating Disk Reactor (although any reactor style should work to some level)
    - Close spaced reactor
- SMI reactor can do
  - LiNbO3 deposition rate high enough to be commercially viable
  - thickness is sufficient for waveguide devices,
    - greater than communications wavelength of 1.55 um
  - can do other compositions, including additions such as K, Ta, V, Ti, Fe and any element having a precursor or is evaporable (ie vapor pressures $\geq$ 0.1 Torr at $\leq$ 350°C)
  - can do doped films, to raise or lower n
  - can deposit films either amorphous or crystalline
  - can deposit other alloys
  - can be used to anneal the films in-situ

Run conditions

*Precursor and cocktail*

Precursors must contain the metals of interest (lithium, niobium, and any dopants such as titanium), be sufficiently volatile (vapor pressure at ~~least~~ 1 Torr at ~~200~~ degrees C), be sufficiently stable (not thermally decompose at the temperature of volatilization), be capable of decomposing to the desired oxide with no unwanted impurities, either with or without an oxidant gas, and be soluble in a suitable solvent. Examples include niobium penta-ethoxide, lithium tert-butoxide, and titanium iso-propoxide but many others are possible.

Solvents must dissolve the precursors at a suitable concentration (being capable of dissolving to the total metal concentration of 0.01 M) and not react with them to form involatile species. Solvents must also have sufficient vapor pressure to be fully vaporized in the flash evaporator at the conditions used for the precursors. Examples include toluene, tetrahydrofuran, alcohols, and many others. In general, these solvents must be kept free of water and other potentially reactive species.

The cocktail should be made up in the absence of water to avoid prereaction of the precursors. The concentration of precursors in solution may range from 0.01 to 1 M; with values in the range 0.05 to 0.2 being especially favorable. Lower concentrations may lead to excessively slow deposition while high concentrations can in some cases lead to inefficient growth. This concentration is a variable to be optimized in each specific set of process conditions.

The feed rate of the cocktail into the flash evaporator can range from ~~0.5 to 1~~0 cc per minute. In practice, rates from 1.3 to 2.5 cc per minute were found to give good results. Too low a rate can lead to drying of the precursor in the lines and clogging (as well as low growth rates), while too high a rate can lead to pooling of solution in the flash evaporator due to inability to volatilize the solution at a fast enough rate (which can give rise to spitting of unvolatilized material through the lines, uneven back pressure, and other unwanted conditions).

*Chamber parameters*

The following range of run parameters can be used to deposit films

| Run parameters | |
|---|---|
| Substrate temperature | 300 - 900° C |
| FE temperature | 200 - 350° C* |
| Chamber pressure | 1 - 100 Torr |
| Uniform gas (Ar or $N_2$) | 500 - 3000 sccm |
| Uniform $O_2$ | 500 - 5000 sccm |
| FE push (inert gas) | 50 - 200 sccm |
| Sample rotation | 500 – 1000 rpm |

Ranges which have been found to deposit good films are:

| Run parameters | |
|---|---|
| Substrate temperature | 400 - 625° C |
| FE temperature | 230° C |
| Chamber pressure | 5 - 20 Torr |
| Uniform inert gas | 500 - 3000 sccm |
| Uniform $O_2$ | 1000 - 5000 sccm |
| FE push (inert gas) | 150 - 200 sccm |
| Sample rotation | 750 |

Amorphous material was found to be deposited at lower temperatures, up to ~450°C substrate temperature. At temperatures of ~475°C or above, the films deposited as crystalline. The films were found to deposit in oriented or epitaxial habit on substrates such as $LiNbO_3$, sapphire, and $LaNiO_3$ and in random orientation on $SiO_2$ substrates.

For the gases used during deposition, the ones above are given as examples but others are possible. The inert gas can be Ar, $N_2$, or other unreactive gas. The oxidant is typically oxygen but can alternatively be $H_2O$, $N_2O$, alcohols such as $CH_3OH$, or others. Good results were obtained with the above conditions but the use of enhancements to the MOCVD deposition process – such as plasma, ultraviolet radiation, or other energetic enhancement, can be anticipated to be useful in some circumstances such as if enhanced deposition rate at low temperatures is desired. In addition, plasma or UV light may be used to clean the substrates prior to deposition.

Amorphous $LiNbO_3$ can be advantageous in that it is easily etchable in solutions such as aqueous HF whereas crystalline $LiNbO_3$ is difficult to etch. Thus the deposition of amorphous $LiNbO_3$ is useful in the creation of patterned devices.

Same as all before, but for K Li Nb Ta O

Electro-optic can include any pyroelectric, ferroelectric and other state

4/4/13

Title: A Scaleable Method for Fabricating Nonlinear Photonic Crystals for Ultrafast All-optical and Electro-optic Functions

**Abstract:**

The University of Wisconsin has developed an MOCVD process by which micron-scale and nanoscale high contrast (air/$LiNbO_3$) epitaxial film photonic structures can be fabricated. As a consequence of the large index difference, as well as the large second order optical nonlinearity of $LiNbO_3$, very compact all-optical and electro-optic devices based on index-confined and photonic bandgap defect waveguides can be realized. These structures will serve as the basis for a new class of truly compact electro-optic and all-optical devices and circuits. The goal of the first phase of this proposed work is to design and demonstrate a reproducible wafer-scale version of the growth process. The follow-on effort will be dedicated to the design, fabrication, and characterization of several devices, including an ultrafast traveling wave modulator, tunable resonator filter, and an all-optical logic switch. This effort will combine the unique expertise of the group of Dr. Leon McCaughan at University of Wisconsin-Madison, discoverer of the technology, Structured Materials Industries, Inc., with extensive knowledge and background in oxide MOCVD processes and equipment, and Pandanus Optical Technologies, for design and fabrication of advanced optical devices. This proposal is addressed to the expressed Air Force need for advanced nanophotonic devices and technology.

Commercial Applications: Successful completion of this program will enable a breakthrough in device capability and quality for a variety of commercial and military applications, including all-optical logic switches, tunable resonator filters and demultiplexers, and very low voltage traveling wave modulators - all part of a growing >$10B market.

KEYWORDS: Lithium niobate, MOCVD, photonic crystal, photonic bandgap, nanophotonics, optical nanodevices, optical switching, modulators

## C. Identification and Significance of the Problem or Opportunity

Photonic crystals, structures in which the refractive index is a periodic function in space, show exciting promise for the realization of a variety of novel and improved optical devices, including single-mode LEDs and thresholdless lasers.[1] $LiNbO_3$, with its large index difference and large second order optical nonlinearity, has the potential to significantly extend the functionality of photonic crystal structures to the all-optical and the ultrafast electro-optical regimes – at the sub-mm scale. Device functions include all-optical logic switches, tunable resonator filters and demultiplexers, and very low voltage traveling wave modulators A major factor inhibiting these applications, however, has been a lack of ability to process and fabricate important optical materials into the complex structures required.

$LiNbO_3$ has posed an especially difficult materials processing problem. $LiNbO_3$ is the nonlinear material of choice for performing all-optical and electro-optic functions. Unfortunately the high chemical stability of crystalline $LiNbO_3$ effectively precludes the use of standard photolithographic patterning techniques: wet etching ($HF/HNO_3$) is limited to several nm/min; dry etching (RIE or RIBE) to 10's of nm/min. The tool set available for processing bulk $LiNbO_3$ is limited to rudimentary functions (e.g., thermal diffusion) on bulk materials. As a consequence, it has not been possible to take advantage of the large refractive index and large nonlinearity of $LiNbO_3$: demonstrated devices are currently based on weakly guiding waveguide-based devices. The successful development of high-index-contrast waveguides and photonic crystal features in nonlinear optical ferroelectrics such as $LiNbO_3$ would serve as the basis for a new class of truly compact electro-optic and all-optical devices and circuits.

Recent results from the McCaughan group at the University of Wisconsin-Madison (UWM) have demonstrated a unique solution to the intractability of crystalline $LiNbO_3$: (1) deposition (via MOCVD) of an amorphous form of $LiNbO_3$, which can be successfully etched, on a crystalline $LiNbO_3$ substrate; (2) etching the amorphous material to form 2-D photonic crystal structures; and (3) annealing the structures to form epitaxial $LiNbO_3$. This work shows tremendous promise of a breakthrough in devices for all-optical signal processing. However, several challenges remain. First, the deposition rate for amorphous $LiNbO_3$ must be increased substantially for practical fabrication of waveguiding layers, and the process must be scaled to full-wafer size to enable commercial scale development.

In this Phase I STTR program, we propose to demonstrate the feasibility of $LiNbO_3$ photonic crystal optical devices on a wafer scale, by combining the strengths of UWM and Structured Materials Industries (SMI). SMI has a long history of developing oxide MOCVD film technology and deposition tools. The Phase I objectives will be to improve deposition rate, taking advantage of recent fundamental mechanistic studies[2] at UWM; modify SMI deposition equipment for the process and transfer the deposition technology to SMI, to show that the process can be carried out in a production scale MOCVD reactor technology; and demonstrate the patternability and optical quality of wafer-scale $LiNbO_3$ epitaxial films. We will also design the commercial $LiNbO_3$ film deposition system. In Phase II, we will build the commercial film deposition system and design, fabricate and characterize several photonic crystal structure devices, including compact, low voltage integrated optical switches and electro-optical modulators. Phase III will consist of system sales and licensed device production.

Successful completion of this program will enable a breakthrough in device capability and quality for a variety of commercial and military applications. As a specific example, the ability to perform intelligent surveillance and target acquisition, to rapidly deploy and process information, and to provide secure command and control communications, is requiring significantly more

rapid and sophisticated data routing and processing than is currently available electronically. Further, large scale long duration satellites are moving to use of all-optical communication networks. The optical devices thus far demonstrated have proven to be impractical for reasons of size, efficiency, speed, and noise. All-optical and electro-optic functions based in LiNbO3 photonic crystal geometries address these deficiencies through reduced size, strong optical confinement, and a large optical nonlinearity. This proposal is addressed to the need for the growth and fabrication of submicron dimensioned photonic heterostructure devices with high dimensional and morphological control in order to enable the monolithic integration of microelectronic and photonic circuits expressed in topic AF02T017, "Nanophotonics".

## C.1. Device objectives

*Ultrafast all-optical signal processing*: Real time optical processing such as packet reading/re-direction for fiber optic systems requires real time logic processes – which in turn requires an optical nonlinearity with no latency. As an elementary example, the AND operation can be formed from the multiplication of two binary (0/1) coded optical signals, $E_j\exp i(\omega_j t)$, j=1,2. The product, taken as either an optical difference- or sum-frequency mixing, is encoded at either the microwave, $E_1E_2\exp i(\omega_1-\omega_2)t$ or optical $E_1E_2 ei(\omega_1+\omega_2)t$ basebands. These two operations can be easily produced by way of one of $LiNbO_3$'s second order nonlinearities. However, at reasonable optical powers, conversion lengths are now ~ several cm because of the large cross section of standard $LiNbO_3$ (diffused) waveguides (~100$\mu m^2$). Both high-contrast index waveguides and photonic crystal defect waveguides, with their inherently small cross sections (~ 0.2 $\mu m^2$), can dramatically reduced the required device length: assuming comparable launched power and a recognizing that the nonlinear conversion efficiency goes as $\eta \sim L^2/A_{eff}$, the conversion length is reduced some 20-fold. In addition, a properly designed resonator can be used to further enhance the nonlinear transition over the intrinsic scattering in the cavity (determined by its Q). Fig. 1 is a schematic diagram of an optical AND element, consisting of a pair of intersecting defect waveguides with a defect cavity at their intersection. A typical scattering lifetime is ~ 5ps, corresponding to a Q ~ $10^3$. Assuming that the energy inside the cavity is ~ 1nJ, the nonlinear decay time is some 300 times faster (more efficient) than the linear scattering process. In the next section, we describe a method for producing 2D photonic crystals in a nonlinear material with the required index contrast (~2:1).

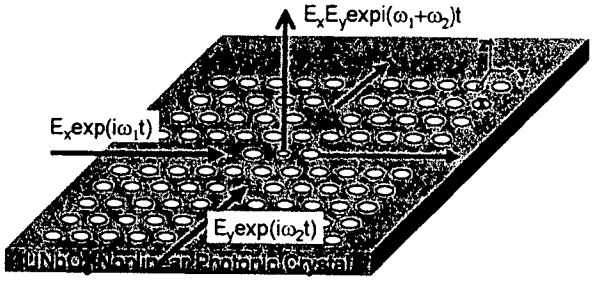


Figure 1. Schematic representation of intersecting defect waveguides in a nonlinear photonic crystal. The d31 coefficient provides the sum frequency product (an optical AND function).

2. *Compact, low voltage integrated optic switches and modulators*: Present-day $LiNbO_3$ integrated optic devices have characteristic lengths ~ several cm, due to the weak confinement of guided light and the consequent large radii of curvature required for device geometries. Using the two-step thin films method we have developed (see below), advantage can be taken of the inherently large refractive index of $LiNbO_3$ to fabricate sub-mm single mode waveguide bends and offsets. The large index contrast, which causes strong optical field confinement, also produces a large microwave field confinement. This combined optical/electrical confinement results in a significant reduction in the operating voltage of electro-optic devices such as traveling wave modulators and optical switch arrays.

We plan to work with device manufacturers such as Pandanus Optical Technologies, Madison WI, and JDS Uniphase (with their worldwide distribution capabilities) to develop both all-optical logic functions as well as low-voltage, ultrafast electro-optic devices such as traveling wave modulators and electro-optically tunable filters.

## C.2 Present Technology Status

*Two-stage growth method*: We have developed a two-stage growth method for fabricating patterned crystalline $LiNbO_3$ structures for photonic crystal and high contrast index waveguide devices, and other photonic applications [patent applied for]. The method uses atmospheric chemical vapor deposition (CVD) to produce an amorphous $LiNbO_3$ film. The film can be patterned using conventional photolithography and standard wet or dry etching techniques, with the crystalline $LiNbO_3$ substrate serving as an etch stop. Alternatively, a standard lift-off process, using $SiO_2$ as a masking material, can be used to produce a desired pattern. When grown on $LiNbO_3$ substrates, a post-growth anneal converts the amorphous film to single crystal $LiNbO_3$. Figure 3 is a cross-sectional TEM image of an amorphous $LiNbO_3$ film after annealing for 1 hour at 1100°C. The inset is the corresponding [01$\bar{1}$0] zone axis selected area diffraction pattern taken from the film/substrate interface area in the image, demonstrating the single crystal epitaxial nature of the layer. The inclined lines are bend contours; the horizontal band is a thickness fringe.

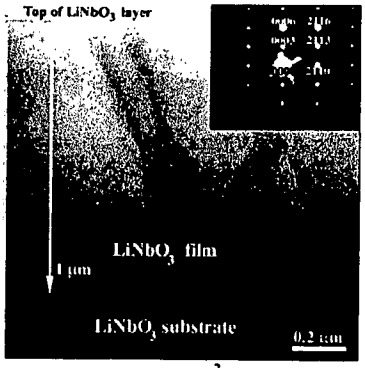



**Figure 2**. TEM of $LiNbO_3$ film[3] after annealing.

As a preliminary feasibility experiment[3], we grew a thin (~ 2 um) amorphous $LiNbO_3$ film on a z-cut $LiNbO_3$ substrate, patterned it with an orthorhombic 2D periodic pattern (7.6 x 10.1 $\mu m^2$), etched in a dilute HF solution, and annealed at 1000°C (Fig. 3). Although not of appropriate dimensions for a linear photonic crystal (~ 0.5 x 0.5 $\mu m^2$ needed), the periodicity is appropriate for phase-matched nonlinear functions.

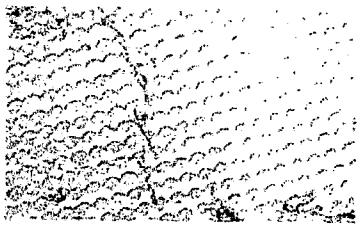


**Figure 3**. Phase contrast micrograph of an orthorhombic lattice (7.6 x 10.1 $\mu m^2$) patterned in $LiNbO_3$

*Enhanced deposition rate:* A major impediment to the exploitation of $LiNbO_3$ thin films is the consistently low growth rates observed for films grown by means of chemical vapor deposition (CVD) or chemical beam epitaxy (CBE). In the course of our thin film work, we identified what we believe is the major source of the very low deposition rates observed with the commonly-used alkoxides precursors: an autocatalytic cycle involving hydrolysis and dehydration which generates volatile monomers of Li and Nb, instead of stable oxides of the metal.[4,5] Figure 4 shows the cycle for $Li(OBu^t)$. In the absence of this cycle, we estimate growth rates would be some 5-10 times larger than the commonly-observed ~ 0.2 μm/h rate. Film thickness, and therefore growth rate, is a critical issue for photonic devices, since for most applications film dimensions must be greater than, or to the order of, the wavelength of the propagated light ($\lambda \geq 1.5\mu m$).

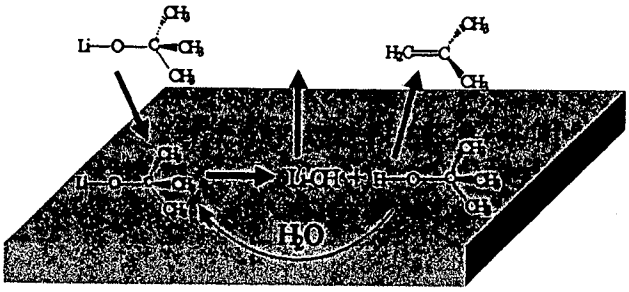


**Figure 4.** Model for the coupled hydrolysis and dehydration of lithium t-butoxide.

The key to overcoming this defect is to redesign the precursors such that they are either more stable to the presence of water, or decompose by a mechanism that removes the elements of water from the surface before dehydration takes place. In principle, these next-generation precursors can be generated by either of two methodologies: a reactive carrier gas (e.g, $Me_3SiCl$) reacts to form more reactive precursor intermediates at the growing surface. Alternatively, and more directly, these precursor intermediates can be prepared in the laboratory and then introduced into the growth introduced directly into the growth chamber. Both of these strategies will be tried in this program to find the best alternative for enhancing the $LiNbO_3$ deposition rate.

Finally, it should be noted that we are not limited to growth of congruent ([Li]/[Nb]=0.94) $LiNbO_3$, as in the case of the bulk material. An amorphous form of stoichiometric $LiNbO_3$ should be straightforward to deposit and anneal to crystallinity. Stoichiometric $LiNbO_3$ is known to have significantly smaller susceptibility to the photorefractive effect (i.e., an increase in the material's refractive index with exposure to visible light), and a five-fold lower voltage required for poling (i.e., re-aligning the ferroelectric domains).

## C.4 SMI Background MOCVD Technology

At SMI, we have historically used a Low Pressure - Rotating Disk Reactor Metal Organic Chemical Vapor Deposition (LP-RDR-MOCVD) system technology for oxide film deposition. We have recently used this system to produce thin films of $Al_2O_3$, $Cu_2O$, $SiO_2$, MgO, $HfO_2$, ZnO, InO, ZnO:Ga/In/Al/B/F (transparent-conductive), SrBiTaO, PbZrTiO, $CeO_2$, $CeMnO_3$, $BaTiO_2$, $SrTiO_2$, and $Ba_xSr_yTiO_2$ (dielectric and pyroelectric); ZnO:Zn, $Zn_2GeO_4$, and $Zn_2SiO_4$:Mn. The system technology has previously been used to produce YBCO, YZO, and an array of other oxides, metals, semiconductors, and so on. Uniform films (±5% thickness uniformity or better) over wafers up to 8" in diameter have been achieved, and a system for coating 12" wafers is under construction. Our system technology has great flexibility in producing a wide range of materials, from dopant levels to alloy levels. Importantly, in addition to demonstrating results with Zn, Si, Ga, Al, Mn, In, B, Sr, Ti, Ce, Bi, Sr, Ta, Pb, Zr, Y, Ba, Cu, Hf, and Mg; this system is also well capable of transporting reactants for films containing Nb, La, Ca, Eu, Er, and a host of other materials, i.e., any compound that can be evaporated. Figure 5 shows a schematic diagram of a SMI RDR reactor system. SMI's specialty is working with customers and collaborators to design and construct MOCVD systems tailored to specific oxide film applications. It is this technology platform that SMI brings to the STTR program to design a commercial scale deposition system for high-rate $LiNbO_3$ deposition.

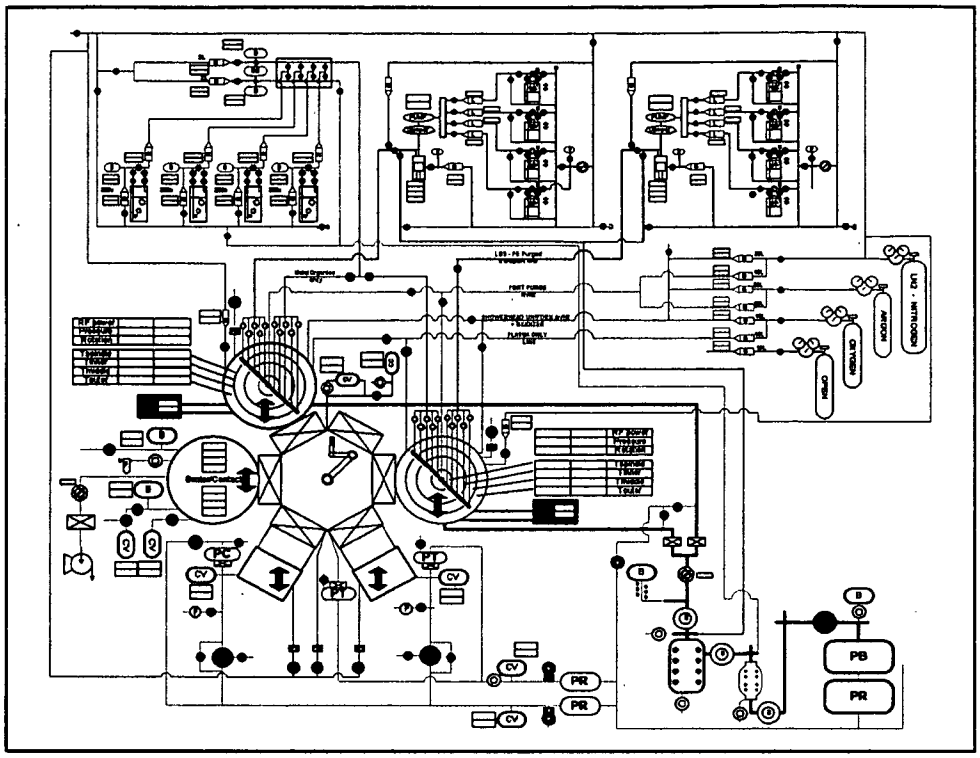



**Figure 5.** Example configuration for SMI multiple source RDR-MOCVD system. (Note: conventional bubbler, flash evaporators or gas sources may be used in this system.).

### C.5. Importance of the STTR Team

The investigators are uniquely suited to carry out this program. A large portion of McCaughan's group at UWM has been devoted to fabrication and characterization of $LiNbO_3$-based nonlinear optics and integrated optics. This group was the first to demonstrate the use of nonlinear photonic crystals for telecommunications applications. Kuech, Saulys, and McCaughan have collaborated extensively in the areas of periodic poling mechanisms and $LiNbO_3$ film growth. Kuech is an internationally known expert both in chemical vapor deposition chemistry and reactor design. The elucidation of the film growth mechanism by Saulys and co-workers have led to the design and synthesis of more effective precursors and reactive carrier gases; these developments have enhanced $LiNbO_3$ thin film growth to the point of enabling growth of prototype device structures. In addition to publications on this collaborative work, the University of Wisconsin has filed provisional patents and plans additional patents.

SMI specializes in process and tool development for commercial scale oxide MOCVD. Dr. Rice is presently leading oxide film growth in ferroelectric oxides such as PZT, PLZT, and CMO, among other oxide MOCVD projects, and Dr. Tompa oversees film growth and directs MOCVD tool development (and has extensive experience with MOCVD of metal oxides and compound semiconductors). No other company has done

as much to advance the state of the art of complex oxide film growth for high technology applications. SMI is thus the ideal partner to work with UWM to produce $LiNbO_3$ films reliably and economically and to develop a tool for commercial production.

Pandanus Optical Technologies of Madison WI will license and commercialize the resulting photonic technologies developed under this effort. JDS Uniphase is also poised to commercialize program results.

## D. Phase I Technical Objectives

The overall technical goal of this Phase I program is to demonstrate the feasibility of producing high quality lithium niobate photonic crystal structures at wafer scale, and to design a high throughput deposition tool for commercial scale production. To fulfill this goal we have the following technical objectives:

| *Objective* | *Description* | *Time Period* |
|---|---|---|
| 1 | Transfer UWM deposition technology to SMI | Months 1 - 2 |
| 2 | Optimize amorphous $LiNbO_3$ deposition using SMI scalable test MOCVD reactor. | 3 - 6 |
| 3 | Demonstrate processability of films | 7 |
| 4 | Refine annealing process for photonic crystal structures | 7 - 8 |
| 5 | Perform chemical and optical characterization of films to determine their suitability for guided wave, electro-optic and photonic bandgap devices. | 8 - 10 |
| 6 | Design commercial scale MOCVD system dedicated to $LiNbO_3$ process, plan Phase II program | 11 - 12 |
| 7 | Write and deliver final report | 12 |

## E. Phase I Work Plan

### E.1 Task 1: Transfer UWM deposition technology to SMI

Drs. Kuech, McCaughan, and Saulys of the University of Wisconsin will work with SMI engineers to design the delivery system for the alkoxides and ethoxide precursors. The SMI scalable research MOCVD reactor will be modified for the specific temperature, pressure, and chemical handling requirements of the UWM process. In particular, some system parts will need to be retooled to be compatible with the chlorine-containing precursors or enhancement agents to be used. UWM and SMI personnel will work together to adapt the deposition process to the rotating disk reactor (RDR) configuration. The UWM process will be demonstrated in the SMI MOCVD system.

### E.2 Task 2: Optimize $LiNbO_3$ Deposition

The objective of this task is to enhance the $LiNbO_3$ deposition rate by incorporating improvements delineated by UWM researchers in their fundamental mechanistic study of the deposition process. This study demonstrated that desorption of Li and Nb species was

autocatalyzed by water released by the decomposition of the alkoxide precursors used. Two potential solutions to this problem were identified: addition of a small quantity of trimethylsilylchloride ($Me_3SiCl$) during deposition using standard alkoxides, or utilizing new precursors designed at UWM. These will be synthesized at UWM and tested as precursors in their own right. The goal is to grow amorphous $LiNbO_3$ at minimum rate of 5 μm/h. Both congruent and stoichiometric thin film compositions will be examined for relative compositional stability. A range of pressure, temperature and flow conditions will be explored to determine the optimum deposition conditions.

### E.3 Task 3: Demonstrate processability

In this task, samples of amorphous $LiNbO_3$ films prepared in Task 2 will be tested for etching rates and quality of etched structures. It will be important to determine if the material prepared under enhanced deposition conditions will behave as expected based on previous experience, and to develop procedures for fabricating device structures.

### E.4 Task 4: Optimize annealing process

The Phase I efforts will continue to refine the annealing process initiated by UWM in order to define manufacturing needs. Because $LiNbO_3$ is subject to $Li_2O$ outdiffusion during high temperature anneals, it may be necessary to design an annealing station which contains an overpressure of $Li_2O$ during the 1100° C annealing process for Phase II. This may be possible to accomplish by annealing the samples in an atmosphere created by an excess of $LiNbO_3$ powder; this is the simplest technique, and ensures that excessive partial pressures of $Li_2O$ are not present. If this is not sufficient to prevent outdiffusion, a more complex annealing station will be designed and built.

### E.5 Task 5: Chemical and optical characterization

Chemical characterization will be performed using SIMS. Structural characterization will be made principally via X-ray diffraction, with a limited number of TEMs to determine defect density. Maker Fringe analysis (a form of surface second harmonic generation) will be used to determine and monitor the Li/Nb ratio. Optical characterization will consist of fabricating channel waveguides and making propagation loss measurements, and determining the magnitude of the nonlinear optic coefficients via the electro-optic effect (e.g., using a standard Mach Zhender waveguide interferometer). We will perform preliminary nonlinear photonic crystal fabrication using electron beam writing capabilities housed within the UW Center for Nanotechnology.

### E.6 Task 6: Design MOCVD system and plan Phase II

The objective of this task is to utilize the results of the Phase I study to design a dedicated MOCVD reactor for the deposition of $LiNbO_3$ films and understand processing issues in a manufacturing environment. The Phase II program will also be planned in detail. A tentative outline of tasks anticipated for Phase II is:

1. Build pilot scale reactor system at SMI for $LiNbO_3$ thin film deposition.
2. Optimize epitaxial deposition for 4-inch wafers with characteristics suitable for integrated optic and photonic crystal applications.
3. Optimize annealing of large area wafers.
4. Chemical and optical characterization of uniformity of crystallized thin films.

41415 MDA/ONR

**Small Busin ss Technology Transfer (STTR) Program**
**Proposal Cover Sheet**

**Propo al Number:** **B023-0099** **Agency: BMDO** DUNS: 787147807

**Topic Number:** **BMDO02T-00** CAGE: OU100

Proposal Title: MOCVD System for LiNbO3 Thin Film Waveguide Modulators and Optical Switches

**Firm:**

Firm Name: Structured Materials Industries

Mail Address: 120 Centennial Ave.

Piscataway, New Jersey 08854-3908

Website Address: www.structuredmaterials.com

Percentage of work: 53 %

**Research Institution:**

Name: University of Wisconsin - Madison

Mail Address: Dept. of Elect. & Comp. Eng.
1415 Engineering Drive
Madison, Wisconsin 53706

Website Address:

Percentage of work: 47 %

Proposed Cost: 70000 Phase: I Duration: 6

**Business Certification:** (Check all that apply)

| | |
|---|---|
| Are you a small business as described in paragraph 2.3? | **YES** |
| Number of employees including all affiliates (average for preceding 12 months): | **12** |
| Is the INSTITUTION a research institute as defined in paragraph 2.4? **University** | **YES** |
| Are you a socially or economically disadvantaged business as defined in paragraph 2.5? | **NO** |
| Are you a woman-owned small business as described in paragraph 2.6? | **NO** |
| Has this proposal been submitted to other US government agencies, or DoD components? | **NO** |

If yes, list the name(s) of the agency or component and Topic Number in the space below.

**Project Manager/Principal Investigator**
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Proprietary Information:

Signature of Principal Investigator Date Corporate Business Official Date Institution Official

**Technical Abstract** (Limit your abstract to 200 words with no classified or proprietary information)

Electro-optical modulators and switches are needed for increased speed, capacity and flexibility of modern optical communications systems. The designs for these devices exist, as do materials with suitable electro optical properties, such as LiNbO3. However, their potential has not been realized, due to the limitations of diffused structures in bulk LiNbO3 crystals. Recently, our STTR partner at The University of Wisconsin - Madison (UWM) have demonstrated that high quality epitaxial LiNbO3 thin films can be produced by MOCVD. The UWM team has also invented a simple process for defining patterned structures from these films. This technology opens the way for a new class of electro-optical devices, including compact high-speed modulators and optical switches.

Structured Materials Industries, Inc. (SMI) has a long history developing MOCVD systems for complex oxide films. UWM will work with SMI to transition the epitaxial LiNbO3 film technology to commercial viability. We will also partner with a commercial supplier of electro-optical components, to provide technical guidance to the Phase I/II efforts and commercialize the resulting products in Phase III. Together, this team is well positioned to commercialize LiNbO3 thin film waveguide devices. UWM has invented the needed process technology and SMI will develop the necessary commercial hardware.

**Anticipated Benefits/Potential Commercial Applications of the Research or Development.** (No classified or proprietary information)

five-fold lower voltage required for poling. We should also be able to obtain engineered doping profiles (e.g. with $Er^{3+}$) potentially leading to a totally new class of electro-optical devices.

### C.4 Device Applications

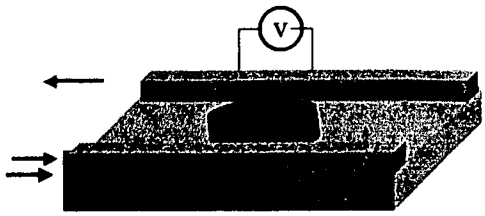


Figure 4: Tunable Ridge Waveguide Ring Resonator

Using the two-step film deposition method, advantage can be taken of the inherently large refractive index of $LiNbO_3$ to fabricate sub-mm single mode waveguide bends and offsets. The large index contrast, which causes strong optical field confinement, also produces a large microwave field confinement. This combined optical/electrical confinement results in a significant reduction in the operating voltage of electro-optic devices, such as traveling wave modulators and optical switch arrays. We plan to work with device manufacturers to develop ultrafast electro-optic devices such as traveling wave modulators and electro-optically tunable filters. The latter is shown schematically in Figure 4. This device is based on high aspect ratio, tightly confining ridge waveguides, which should be easily fabricated using the UWM two stage process. In Phase I, we will demonstrate the feasibility of producing these devices. In Phase II, we will design and fabricate compact, low voltage integrated optic switches and modulators using this technology.

### C.5 Importance of the STTR Team

The investigators are uniquely suited to accomplish the goals of this program. McCaughan's group at UWM has been devoted to fabrication and characterization of $LiNbO_3$ based nonlinear optics and integrated optics. This group was the first to demonstrate the use of nonlinear photonic crystals for telecommunications applications. Kuech, Saulys, and McCaughan have collaborated extensively in the areas of periodic poling mechanisms and $LiNbO_3$ film growth. Kuech is an internationally known expert both in CVD processes and reactor design. The elucidation of the film growth mechanism by Saulys and co-workers has led to the design and synthesis of more effective precursors and reactive carrier gases.

SMI is the leading US supplier of MOCVD systems for complex oxide thin films. SMI is currently developing fully integrated systems for Rotating Disc Reactor - Metal-Organic Chemical Vapor Deposition (RDR-MOCVD). We have used this technology to produce thin films and multilayers of a wide variety of complex oxide materials. Of particular interest to this effort is our related work on perovskite materials, such as $BaTiO_3$, $SrTiO_3$, $Ba_xSr_yTiO_2$ $SrBi_2TaO_9$ and $PbZr_xTi_{1-x}O_3$. Refer to Section F.

Pandanus Corporation was founded in 2001 to develop and commercialize the groundbreaking $LiNbO_3$ optical device technologies that originated at the University of Wisconsin. Pandanus original market studies indicate the potential for $500 million in component sales if fundamental $LiNbO_3$ device design limitations could be overcome. Pandanus is also building relationships with companies such as JDS Uniphase, to help with the insertion of these devices into today's fiber-optic telecommunication market. Pandanus Optical Technologies will license and commercialize the resulting photonic products developed under this effort.

### C.6 Bibliography

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## *D. Phase I Technical Objectives:*

The mission for the UWM / SMI team is to bring the UWM process technology to market. We will accomplish this in two steps. In Phase I, we will demonstrate technical feasibility. In Phase II, we will develop actual products. We will also partner with commercial organizations engaged in the development of fiber optic communications products. Pandanus Optical Technologies, of Madison, WI will provide technical input to the initial stages of this effort, and license and commercialize the resulting electro-optical devices during later program stages.

The UWM / SMI / Pandanus team is uniquely qualified to accomplish the goals of this program. Each brings specific capabilities to the effort. UWM provides the thin film process technology, and materials and device characterizations capabilities. SMI brings expertise in development and commercialization of MOCVD film deposition systems. Pandanus provides knowledge of optical communications technology and products. In addition, we will also partner with JDS Uniphase, who brings worldwide distribution capabilities to this effort.

The following Table summarizes the team's objectives for the Phase I program, as well as for Phase II and Phase III.

| **Phase I Objectives** | |
|---|---|
| 1 | Identify critical hardware issues for the UWM MOCVD process and develop specifications for a commercial epitaxial $LiNbO_3$ film deposition system. |
| 2 | Demonstrate properties of the resulting epitaxial films that meet the requirements for compact, high-speed electro-optical devices. The Phase I targets are epitaxial $LiNbO_3$ films, at least 2 microns thick, deposited at rates of at least 1 micron/hour, with optical losses of 1 dB/cm or less. |
| **Phase II Objectives** | |
| 1 | Develop a production worthy MOCVD system for deposition of epitaxial $LiNbO_3$ films, based on the UWM technology. The system will meet all process specifications established in Phase I, as well as ultimate customer expectations for reliability and economical operation. |
| 2 | Develop and refine the $LiNbO_3$ MOCVD deposition process, to produce films suitable for electro-optic device fabrication, on wafer sizes up to six inches. The Phase II targets are epitaxial $LiNbO_3$ films, greater than 5 microns thick, deposited at rates of 5 micron/hour or better, with optical losses less than 1 dB/cm. |
| 3 | Develop and demonstrate $LiNbO_3$ thin film waveguide devices, including compact high-speed electro-optical modulators and optical switches. Develop strategic partnerships with device manufacturers for licensing and commercialization of the devices. |
| **Phase III Objectives** | |
| 1 | Commercialize the complete thin film deposition system. |
| 2 | Commercialize $LiNbO_3$ based electro-optical modulators and optical switches through strategic partnerships. |

## *E. Phase I Work Plan:*

| *Task Number* | *Task Description* | *Month* |
|---|---|---|
| 1 | Transfer the UWM process technology to development MOCVD reactor at SMI. | 1 - 3 |
| 2 | Optimize the MOCVD precursor chemistry for maximum deposition rate and consistent process performance. | 2 - 4 |
| 3 | Refine and demonstrate amorphous $LiNbO_3$ deposition using scalable test MOCVD reactor at SMI. | 2 - 4 |
| 4 | Refine and demonstrate annealing technology to produce epitaxial $LiNbO_3$ films from the amorphous deposits. Characterize the films chemical, physical and optical properties and demonstrate their suitability for waveguide devices. | 3 - 5 |
| 5 | Provide films to electro-optical device manufacturers and develop strategy for device implementation. | 4 - 5 |
| 6 | Design scaled up reactor. | 5 |
| 7 | Deliverable - Phase I final report and Phase II proposal. | 6 |

Task 1: Transfer UWM process technology to development MOCVD reactor at SMI.

UWM will establish a list of critical process issues for MOCVD of epitaxial ferroelectric thin films. This list will define the process requirements in terms of substrate temperature, gas flow rates, precursor chemistry, precursor delivery method, atmospheric moisture tolerance, oxygen activation method and other hardware related issues. This information will be established partly through experimentation and partly from existing process technology at UWM. Drs. Kuech, McCaughan, and Saulys of the University of Wisconsin will work with SMI engineers to facilitate all aspects of the technology transfer, including the design of the delivery system for the alkoxide and ethoxide precursors. The deliverable for this task will be the successful transfer of the UWM process to the development reactor at SMI, and a set of specifications for the production MOCVD system for epitaxial $LiNbO_3$ film deposition.

Task 2: Optimize the MOCVD precursor chemistry for maximum deposition rate and consistent process performance.

UWM will continue with their research to determine the mechanisms and kinetics of $LiNbO_3$ deposition from alkoxides and ethoxide precursors. The proposed UWM process takes advantage of their recent findings regarding the unwanted formation of volatile hydroxides. To avoid this unwanted process, a reactive carrier gas such as $Me_3SiCl$ will be investigated. Preliminary finding by the UWM team show that the introduction of a small amount of $Me_3SiCl$ doubles the growth rate of $Nb_2O_5$. Experiments will be done to determine if similar results can be obtained for $LiNbO_3$. In addition, UWM has identified the likely precursor intermediates responsible for this increase in growth rate. These intermediates will be synthesized at UWM and evaluated as precursors in their own right. The deliverable for this task will be an assessment of which approach is best for the MOCVD precursor chemistry.

Task 3: Refine and demonstrate amorphous $LiNbO_3$ deposition using scalable test MOCVD reactor at SMI.

SMI will perform process investigations on the development MOCVD reactor, starting with the UWM process and precursors. We will use low cost substrates (such as silicon or sapphire) for the initial process development. We will then use either $LiNbO_3$ or $LiTaO_3$ single crystal substrate, for demonstration of the

optimized epitaxial film properties. The primary intent of this task will be to demonstrate a process to deposit amorphous $LiNbO_3$ films, at least two microns thick, at growth rates of at least one micron/hour. In the course of this activity, we will identify any hardware issues related to the scale-up of the process to commercial wafer sizes and production volumes. We will show proof of concept that we can resolve all scale-up issues. We will also investigate different $LiNbO_3$ film compositions, including both the congruent ([Li]/[Nb] = 0.94) and stoichiometric ([Li]/[Nb] = 1.00) film compositions. Bulk $LiNbO_3$ materials are limited to the congruent composition. However, the MOCVD process should allow us to deposit films of any composition. Selected films from this task will be provided to UWM for evaluation as described in Tasks 4 and 5 below.

Task 4: Refine and demonstrate annealing technology to produce epitaxial $LiNbO_3$ films from the amorphous deposits. Characterize the films chemical, physical and optical properties and demonstrate their suitability for waveguide devices.

UWM will refine and demonstrate the annealing technology to produce epitaxial $LiNbO_3$ films from the amorphous deposits produced at SMI in Task 3. Because $LiNbO_3$ is subject to $Li_2O$ out-diffusion during high temperature anneals, it may be necessary to design an annealing station which contains an overpressure of $Li_2O$. The goal of this task will be to demonstrate epitaxial films, of 2 micron thickness or greater, without stress related failures such as cracking. We will also demonstrate a process to pattern the amorphous deposits, and anneal them to produce pre-defined structures in the epitaxial $LiNbO_3$ films.

The resulting thin films will be characterized as needed at UWM. Chemical characterization will be made using SIMS. Structural characterization will be made principally via X-ray diffraction, with a limited number of TEMs to determine defect density. Maker Fringe analysis (a form of surface second harmonic generation) will be used to determine and monitor the Li/Nb ratio. These characterization results will be provided as feed-back to the on-going process development work at SMI.

Optical characterization will consist of fabricating channel waveguides and making propagation loss measurements, and determining the magnitude of the nonlinear optic coefficients via the electro-optic effect (e.g. using a standard Mach Zehnder waveguide interferometer). Our targets for this task are to obtain films with optical loss of 1 dB/cm or less, and electro-optic properties approaching that of bulk $LiNbO_3$. UWM is fully equipped to carry out all aspects of this characterization. See Section J for a description of facilities.

Task 5: Provide films to electro-optical device manufacturers and develop strategy for device implementation.

Selected samples of the most promising epitaxial $LiNbO_3$ films will be fabricated into simple waveguide structures and devices at UWM. We will provide samples to our partners at Pandanus Optical Technologies and JDS Uniphase (and other potential customers/partners that we identify) for their evaluation. The deliverable for this task will be verification that the films have the necessary properties for application in electro-optical devices, and a plan for the development of these devices in the Phase II effort.

Task 6: Design scaled up reactor.

Using the results of Tasks 1 through 3, we will establish the design for a commercial MOCVD system, scaling the UWM process technology up to commercial wafer sizes and production volumes. We anticipate that the major design issues will involve the precursor delivery system and the vacuum technology to exclude atmospheric moisture from the deposition chamber. Other engineering considerations in the design include the chamber wall temperature, which must be high enough to prevent precursor condensation but low enough to avoid film deposition. Materials compatibility in the precursor delivery system will also need to be considered. Gas flow effects will need to be evaluated, particularly

buoyancy effects above the disk at the high temperatures. SMI has the engineering expertise, and the computer modeling capabilities (if necessary) to resolve each of these issues. We are well experienced and well qualified in this type of system design. The deliverable for this task will be a set of design specifications for the epitaxial $LiNbO_3$ thin film production system. The actual development of the production system will proceed in Phase II.

Task 7: Reporting.

In accordance with the requirements of the MDA, we will document all of our findings in a final report. If feasibility is demonstrated, we will also present our plans for further development in a Phase II proposal.

Throughout this effort, both SMI and UWM will secure intellectual property rights for the hardware and processes by filing for patents, as appropriate. We will also disseminate technical information through technical publications, conference presentations, trade shows and through our product marketing efforts at SMI.

## *F. Related Work:*

### F.1 Related Research at The University of Wisconsin - Madison

Simultaneous all-optical wavelength interchange: Reported the first telecommunication application of 2-D nonlinear lattices: the theoretical demonstration of simultaneous optical wavelength interchange. The two DFG processes essentially "diffract" the interconverted signals from the unconverted ones, providing spatial segregation to eliminate coherent in band cross talk. Presented the first experimental demonstration of simultaneous optical wavelength interchange between the wavelengths 1535 nm and 1555 nm.

Er:Yb:LiNbO3 waveguide optical amplifier: Developed a method for greatly enhancing the local incorporation of $Er^{3+}$ into Ti:$LiNbO_3$ integrated optic devices. In the course of this work identified 4 major sites of $Er^{3+}$ incorporation into $LiNbO_3$. Demonstrated enhanced pump absorption via $Yb^{3+}$ co-doping. Out of this basic research, demonstrated a 980 nm pumped $LiNbO_3$ waveguide optical amplifier.

A rigorous theory for intersecting optical waveguides: Developed a rigorous theory for intersecting optical waveguides which correctly explains and predicts all of the optical characteristics of this waveguide geometry: excellent agreement between calculated and measured coupling constants is found; prediction and observation of a sharply peaked radiation pattern, which in turn explains the anomalously large losses at certain intersection angles; modification of the refractive index of the region common to intersecting waveguides (fractional doping) provides a mechanism for controlling the guided-guided optical coupling characteristics.

Fundamental limits to crosstalk in Ti:$LiNbO_3$ devices: Predicted and subsequently verified that small, randomly distributed, index variations in optical switches produces crosstalk and an asymmetric response.

Three-electrode geometry reduces optical crosstalk in Ti:$LiNbO_3$ switches. We demonstrated with model calculations and by experiment that a 3-segment electrode would provide sufficient degrees of electrical freedom to compensate for the randomly distributed phase mismatch in optical switches and therefore reduce optical crosstalk. We demonstrated a record extinction ratio of -48 dB, almost two orders of magnitude improvement.

Faster integrated optical devices without a power penalty: Our calculations show that the voltage-length product of directional coupler switches is not a constant of the device, as previously presumed (e.g., less

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## C. *Identification and Significance of the Opportunity:*

### C.1 Overview

To move beyond the current use of fiber optics as a point-to-point "telegraph" system will require the development of high speed electro-optical modulators and optical switches. $LiNbO_3$ is a nearly ideal material for fabrication of these devices, due to its large refractive index, excellent transparency and electro-optical properties. However, the potential of this material has yet to be realized commercially. Few products have been developed to date, due to the lack of a suitable technology to fabricate $LiNbO_3$ thin film waveguide structures.

Recently, our STTR partners at The University of Wisconsin - Madison (UWM) have developed a two step process for producing waveguide structures in epitaxial $LiNbO_3$ thin films. The patented UWM process results in $LiNbO_3$ films of sufficient thickness and excellent quality, suitable for electro-optical device applications. The UWM process also enables high deposition rates, and relatively simple sub-micron patterning, both of which impact commercial viability in a positive way. The resulting waveguide structures will serve as the basis for a new class of truly compact electro-optic devices, offering greater speeds and lower operating voltages.

In this effort, UWM and SMI propose to work together to transfer this technology to commercialization. UWM will provide the thin film technology. SMI will development the deposition equipment. We will also work with potential end-users, including Pandanus Optical Technologies and JDS Uniphase, for the electro-optical device technology and ultimate device commercialization. Together, this team is well positioned to accomplish the objectives of the proposed effort. In Phase I, we will demonstrate proof of concept for a commercial, wafer-scale version of the UWM process. In Phase II, we will build the commercial film deposition system and design, fabricate and characterize electro-optical devices. In Phase III, we will commercialize the film deposition systems and the resulting electro-optical devices.

### C.2 Materials Issues for Electro-optical Devices

Modern communication is increasingly based on fiber optics. This is due to the fact that optical signals carry a higher information content (more bits/second) than conventional electrical signals[1]. To gain even greater capacity, optical networks multiplex many signals, in parallel, over a single fiber, using wavelength division multiplexing (WDM) and dense wavelength division multiplexing (DWDM) technologies. Managing these multiple signals requires compact high-speed, low voltage electro-optical components, such as optical switches and modulators. These components, along with their production scale manufacturing technology, are the focus of this proposal.

Optical waveguides require materials with good transparency at the wavelength of interest, (1.55 microns in current telecommunications systems). Waveguides serve as interconnects in opto-electronic integrated circuits (OEIC's) as well as form the basic components of devices such as optical switches and modulators. Waveguide switches and modulators change optical path length in response to electrical signals. A common example is the Mach-Zehnder interferometer[2]. These components require materials that can change optical properties, in a non-linear fashion, in response to electrical signals. Ferroelectric $LiNbO_3$ is a nearly ideal material for both the OEIC interconnects and electro-optical components, having both excellent transparency and a large electro-optical coefficient.

Present day versions of electro-optical switches and modulators are based on bulk crystals of $LiNbO_3$. A third elemental species (typically titanium) is diffused into the crystal to define waveguide layers in the surface[3]. The concentration profile of these waveguide layers is limited to the typical error-function shaped diffusion profile, and thus only graded index waveguides can be produced. As a consequence of the resulting weak confinement of the guided wave, bulk crystal $LiNbO_3$ devices have characteristic lengths on

the order of several centimeters, and large radii are required for device geometries. The end-result is devices that are large, and consequently slow and require high operating voltages.

Ideally, optical waveguide components would be fabricated from thin films of ferroelectric $LiNbO_3$. A thin film deposition technology, such as metal-organic chemical vapor deposition (MOCVD), would allow fabrication of step index waveguide structures, or any other designed concentration profile. This would provide better confinement of the optical signal, and greater flexibility in the device design. The result would be more-compact devices, with consequent higher speeds, lower operating voltages and greater a degree of device integration.

Previous attempts to make $LiNbO_3$ thin films used sputtering[4,5], laser ablation[6-8], sol-gel processing[9], thermal plasma spray CVD[10,11], liquid phase epitaxy[12,13], chemical beam epitaxy[14] and MOCVD[15-22]. Although there have been many encouraging reports of epitaxial deposition, in general, the films suffer from being too thin and from having excessive optical losses. For effective waveguiding, the films thickness must be on the order of the communication wavelength (presently 1.55 microns). Epitaxial $LiNbO_3$ film deposition on sapphire has only achieved a thickness up to 2000 angstroms, because of cracking caused by the large thermal expansion mismatch with the substrate[17]. $LiTaO_3$ substrates have a much better thermal expansion match with $LiNbO_3$, but have only resulted in films up to 6000 angstroms thick[17]. Effective waveguiding also requires films with very low optical loss. Nominally, losses of less than 1 dB/cm are required. The sources of optical losses in thin films are; scattering by defects in the film, scattering by surface roughness, optical absorption and optical dispersion due to polycrystalline materials[23]. Therefore, low-defect density films are required, with low surface and interfacial roughness. The films must also have high purity and good oxygen stoichiometry (for low optical absorption) and must be single crystalline (for minimum optical dispersion).

Another obstacle to implementing thin film $LiNbO_3$ devices is the lack of an efficient patterning technology. Unfortunately the high chemical stability of crystalline $LiNbO_3$ effectively precludes the use of standard photolithographic patterning techniques. Wet etching ($HF/HNO_3$) is limited to several nm/min. Dry etching (RIE or RIBE) to 10's of nm/min. In fact, the present tool set available for processing bulk $LiNbO_3$ is limited to rudimentary functions (e.g. thermal diffusion) on bulk materials. As a consequence, it has not been possible to take advantage of the large refractive index and large optical nonlinearity of $LiNbO_3$. Demonstrated devices have thus far been based on weakly guiding waveguide structures. The successful development of a technology for deposition and patterning of high quality $LiNbO_3$ thin films would enable a new class of truly compact electro-optic devices and circuits.

### C.3 UWM Two Stage Growth Process

UWM has developed a two-stage growth process for fabricating patterned structures of crystalline $LiNbO_3$ for photonic crystal and electro-optical waveguide devices[24]. The method uses MOCVD to deposit an amorphous $LiNbO_3$ film. The amorphous $LiNbO_3$ layer can be easily patterned using conventional photolithography and standard wet or dry etching techniques. For films deposited on crystalline $LiNbO_3$, the substrate serves as an effective etch stop. For amorphous films grown on $LiNbO_3$ substrates, a post deposition anneal converts the amorphous film to single crystal epitaxial $LiNbO_3$. We assume that similar epitaxial films would be obtained on closely lattice-matched substrates such as $LiTaO_3$.

As a preliminary feasibility experiment, UWM grew a 2 micron thick amorphous $LiNbO_3$ film on a z-cut $LiNbO_3$ substrate. The film was then patterned, with an orthorhombic 2D periodic pattern (7.6 x 10.1 $\mu m^2$), using standard photolithography and wet etching in dilute HF. Subsequent annealing at 1000 C resulted in the crystalline epitaxial $LiNbO_3$ structure shown in Figure 1.

Figure 2 is a cross-sectional TEM image of a similar amorphous $LiNbO_3$ film after annealing for 1 hour at 1100 C. The insert is the corresponding [01-10] zone axis selected area diffraction pattern, taken from the

film/substrate interface area in the image, demonstrating the single crystal epitaxial nature of the layer. The inclined lines are bend contours; the horizontal band is a thickness fringe. **To our knowledge, this is the first time a 2 micron thick epitaxial $LiNbO_3$ film has been demonstrated.** The previous largest thickness reported was 0.6 microns for an epitaxial film on $LiTaO_3$[17]. These results demonstrate the capabilities of the UWM two stage process, namely the ability to make epitaxial $LiNbO_3$ films, thick enough for practical waveguide applications, and the ability to readily pattern these films to fine geometries.

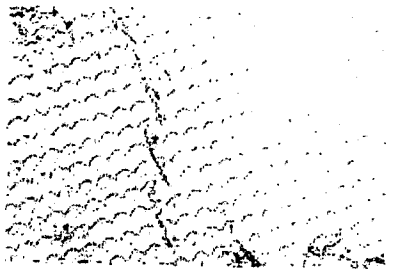

**Figure 1:** Phase contrast Micrograph of an orthorhombic lattice (7.6 x 10.1 $\mu m^2$) patterned in epitaxial $LiNbO_3$.

Another major impediment to the implementation of $LiNbO_3$ thin films is the consistently low growth rates observed for films deposited by MOCVD or chemical beam epitaxy (CBE). Reasonable growth rates are an important consideration for commercial viability. In the course the thin film work at UWM, the researcher identified what they believe is the source of the low deposition rates observed for the commonly used alkoxides precursors[25]. The culprit is an autocatalytic cycle involving hydrolysis and dehydration, which generates volatile monomers of Li and Nb, instead of stable oxides of the metal. Figure 3 shows the cycle for lithium butoxide. In the absence of this cycle, the estimated growth rates for CBE would be some 5 to 10 times larger than the commonly observed; ~ 0.2 um/h rate.

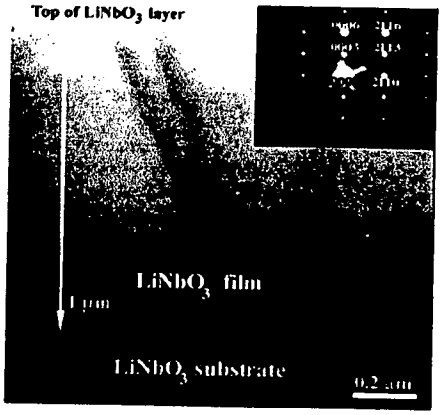


**Figure 2:** TEM of LiNbO3 film after annealing.

The key to overcoming this defect is to redesign the precursors such that they are either more stable to the presence of water, or decompose by a mechanism that removes the elements of water from the surface before dehydration takes place. In principle, these next-generation precursors can be generated by either of two methodologies. A reactive carrier gas (e.g., $Me_3SiCl$) can be used to form more reactive precursor intermediates at the growing surface. Alternatively, and more directly, these precursor intermediates can be prepared in the laboratory and then introduced directly into the deposition chamber.

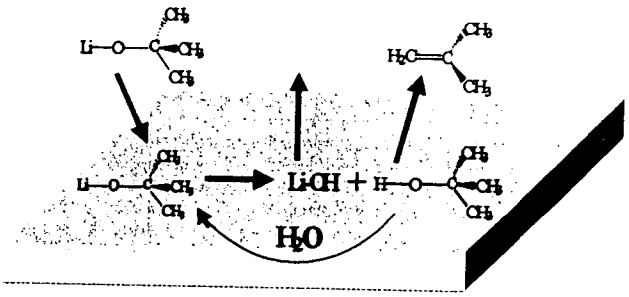


**Figure 3:** Model for the coupled hydrolysis and dehydration of lithium t-butoxide.

The UWM two stage process results in epitaxial $LiNbO_3$ films, of useful film thickness, and the ability to pattern these films to fine geometries. We also believe we can dramatically increase the deposition rate for $LiNbO_3$, and improve the commercial viability of the process. The other potential advantages of the process are those inherent to MOCVD. We can readily control the composition and composition profile of the films. High purity $LiNbO_3$ films should be achievable. (Bulk $LiNbO_3$ crystals frequently have Fe contamination). In addition, we are not limited to growth of the congruent ([Li]/[Nb]=0.94) composition, as in the case of the bulk material. An amorphous form of stoichiometric ([Li]/[Nb]=1.00) $LiNbO_3$ should be straightforward to deposit and anneal to crystallinity. Stoichiometric $LiNbO_3$ is known to have significantly smaller susceptibility to the photorefractive effect (i.e. an increase in the material's refractive index with exposure to visible light) and a

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| Project Manag r/Principal Inv stigator | | Corporat Official (Busine ) | | Institution Official | |
|---|---|---|---|---|---|
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***For any purpose other than to evaluate the proposal, the data referenced below shall not be disclosed outside the Government and shall not be duplicated, used or disclosed in whole or in part, provided that if a contract is awarded to this proposer as a result of or in connection with the submission of this data, the Government shall have the right to duplicate, use or disclose the data to the extent provided in the funding agreement. This restriction does not limit the Government's right to use information contained in the data if it is obtained from another source without restriction. The data subject to this restriction is contained on the pages of the proposal listed on the line below.***

Proprietary Information (list page numbers): 3-25

| Signature of Principal Investigator | Date | Corporate Business Official | Date | Institution Official |
|---|---|---|---|---|

**Technical Abstract** (Limit your abstract to 200 words with no classified or proprietary information)

In this Phase I STTR effort, Structured Materials Industries, Inc. (SMI), in collaboration with our academic partners at the University of Wisconsin at Madison (UWM), will develop technology to build chip-scale integrated photonic crystal device networks. The photonic crystal devices will be fabricated in epitaxial lithium niobate (LiNbO3) thin films. This effort we will build on technology invented at UWM, to deposit and pattern epitaxial LiNbO3 thin films.

In this Phase I effort, we will develop the technology to integrate photonic device structures into chip-scale optical networks. We will demonstrate the integration technology by building and testing an integrated electro-optically gated 4-channel add/drop multiplexer.

In Phase II, we will design and fabricate networks containing different optical and electro-optical devices, including lasers, detectors, switches, modulators and multiplexers. The network components will be connected with photonic crystal waveguides, built directly into the epitaxial LiNbO3 thin film. In Phase III, we will commercialize this technology for both government and private sector markets.

**Anticipated Benefits/Potential Commercial Applications of the Research or Development.** (No classified or proprietary information)

The successful development of this integration technology, combined with our LiNbO3 epitaxial film technology, will enable a direct route to large-scale integrated (LSI) optical device networks. These products will initially find applications in military markets, integrating high-speed communications from command, control and sensor arrays. These products will also meet growing market demands for telecommunications, digital signal processing and all optical computing. These markets are expected to reach multi-billion dollar size by the year 2006.

**List a maximum of 8 Key Words or phrases that describe the Project.**

LiNbO3
Thin Films
Epitaxy
Patterning
Photonic Crystals
Optical Networks
Add/Drop Multiplexer

**Small Business Technology Transfer (STTR) Program**
**Proposal Cover Sheet**

## Proposal Number: F033-0164 Agency: Air Force

DUNS: 787147807
CAGE: OU100

**Topic Number:** AF03T021

**Proposal Title:** Chip-scale Photonic Crystal Optical Networks Based on Epitaxial LiNbO3 Thin Films

### Firm:

**Firm Name:** Structured Materials Industries

**Mail Address:** Suite 103
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**Website Address:** www.structuredmaterials.com

**Percentage of work:** 58 %

### Research Institution:

**Name:** University of Wisconsin

**Mail Address:** 750 University Avenue

Madison, WI 53706

**Website Address:** www.wisc.edu

**Percentage of work:** 42 %

**Proposed Cost:** 100000 **Phase:** I **Duration:** 9

## Business Certification: (Check all that apply)

| | |
|---|---|
| Are you a small business as described in paragraph 2.3 (note: wholly owned subsidiaries are not eligible)? | YES |
| Number of employees including all affiliates (average for preceding 12 months): | 12 |
| Is the INSTITUTION a research institute as defined in section 2.4? paragraph 2.4? | YES |
| University | |
| Ar you a socially or economically disadvantaged business as defined in paragraph 2.5 ? | NO |
| Are you a woman-owned small business as described in paragraph 2.6 ? | NO |
| Are you a certified HUBZone small business concern as described in paragraph 2.12 ? | NO |
| Has this proposal been submitted to other US government agencies, or DoD components? | NO |

If yes, list the name(s) of the agency or component and Topic Number in the space below.

# Chip-scale Photonic Crystal Optical Networks Based on Epitaxial $LiNbO_3$ Thin Films

**STTR Phase I proposal. Submitted in response to Topic:**
AF03T021 "Photonic Crystal Chip-scale Optical Networks"
April, 2003

## *Part 1: Identification and Significance of the Opportunity:*

### 1.1 Overview

In this Phase I STTR effort, Structured Materials Industries, Inc. (SMI), in collaboration with our academic partners at the University of Wisconsin at Madison (UWM), propose to develop technology to build chip-scale integrated photonic crystal device networks. The photonic crystal devices will be fabricated in epitaxial lithium niobate ($LiNbO_3$) thin films, using standard photolithography and reactive ion etching techniques. $LiNbO_3$ is an excellent material for fabricating photonic crystal devices. $LiNbO_3$ has excellent transparency to the wavelength of interest in optical communications and all-optical computing. In addition, its large second order optical nonlinearity enables highly compact photonic crystal devices. Previously, the full potential of $LiNbO_3$ devices has not been exploited, due to the lack of a suitable deposition and patterning technology for $LiNbO_3$ thin films.

In this proposed effort, we will build on technology invented at UWM to deposit and pattern epitaxial $LiNbO_3$ thin films. During the past year, SMI and UWM have worked together to develop and optimize the UWM process technology and related production hardware. We are also working on technology to build photonic device structures in the resulting epitaxial $LiNbO_3$ thin films. In this effort, we plan to build on these accomplishments. In this Phase I effort, we will develop the technology to integrate photonic device structures into chip-scale optical networks. We will demonstrate the integration technology by building an integrated electro-optically gated 4-channel add/drop multiplexer. This highly compact device will have a footprint of approximately 1 $mm^2$. The same device made using diffused waveguides in bulk $LiNbO_3$ crystals would have dimensions on the order of 10's of $cm^2$.

In Phase II, we will further develop and refine the integration technology for $LiNbO_3$ photonic devices. We will design and fabricate networks containing different optical and electro-optical devices, including lasers, detectors, switches, modulators and multiplexers. Many of these components can be constructed directly in the epitaxial $LiNbO_3$ thin film. The network components will be connected with photonic crystal waveguides, also built into the $LiNbO_3$ film. This integration technology, combined with our $LiNbO_3$ epitaxial film technology, will enable a direct route to large-scale integrated (LSI) optical device networks. Also during Phase I and II, we will work with our commercial partners, who include Pandanus Optical Technologies and JDS Uniphase, to plan a commercialization route for the resulting new class of integrated optical devices.

In Phase III, we will design and build integrated optical network devices for military needs. We will also commercialize this technology for both government and private sector markets. We will

commercialize the integrated optical devices through our partners at Pandanus Optical Technologies and JDS Uniphase. Structured Materials Industries will also commercialize the deposition and processing hardware to build these devices.

## 1.2 Background - Photonic Crystal Devices

Photonic crystals are two and three-dimensional periodic refractive index modulations. The appropriate geometry produces photonic bandgaps, which are frequency ranges where the propagation of light is forbidden inside the structure. Strongly confining channel waveguides can be fabricated by introducing a line of defects in the "crystal." These photonic waveguides can be constructed with very sharp bends and with very low losses[1]. Single-mode LEDs and thresholdless lasers are another consequence of a photonic bandgap lattice's ability to alter the spontaneous emission pattern and rate of an emitting atom[2]. We note that the large refractive indices of ferroelectrics will support 2D air/substrate photonic crystal geometries, giving rise to a new class of low-power, easily integrated, all-optical devices and circuits.

Ferroelectric $LiNbO_3$ is a nearly ideal material for photonic crystal devices, due to its large index difference and large second order optical nonlinearity. Unfortunately, $LiNbO_3$ device development has been limited by the lack of suitable technology for deposition and patterning of $LiNbO_3$ thin films. Present day $LiNbO_3$ devices are fabricated using bulk crystals. A third element (typically titanium) is diffused into the crystal to define waveguide layers in the surface[3]. The concentration profile of these waveguide layers is limited to an error-function shaped diffusion profile, and thus only graded index waveguides can be produced. As a consequence of the resulting weak confinement of the guided wave, bulk crystal $LiNbO_3$ devices have characteristic lengths on the order of several centimeters, and large radii are required for device geometries. The end-result is devices that are large, slow and require high operating voltages.

Ideally, photonic crystal structures could be fabricated in epitaxial thin films of ferroelectric $LiNbO_3$. A thin film deposition technology, such as metal-organic chemical vapor deposition (MOCVD), would allow fabrication of step index waveguide structures, or any other designed concentration profile. With the availability of a suitable pattering technology, photonic bandgap structures could be fabricated in epitaxial $LiNbO_3$ thin film. The resulting photonic crystal devices could perform a wide variety of optical functions, including switching, filtering and modulation. Optical interconnects could also be defined in the $LiNbO_3$ flayer. The resulting integrated devices would be more compact, operate at higher speeds and lower voltages, and provide for a greater a degree of device integration.

Previous attempts to make $LiNbO_3$ thin films have used sputtering[4,5], laser ablation[6-8], sol-gel processing[9], thermal plasma spray CVD[10,11], liquid phase epitaxy[12,13], chemical beam epitaxy[14] and MOCVD[15-22]. Although there have been many encouraging reports of epitaxial deposition, in general, the films suffer from being too thin and from having excessive optical losses. Epitaxial $LiNbO_3$ film deposition on sapphire has only achieved a thickness up to 2000 angstroms, because of cracking caused by the large thermal expansion mismatch with the substrate[17]. $LiTaO_3$ substrates have a much better thermal expansion match with $LiNbO_3$, but have only resulted in films up to 6000 angstroms thick[17]. (As descried in Part 4.1 of this proposal, the UWM/SMI team has recently achieved $LiNbO_3$ films up to 3 microns thick on silicon and z-cut $LiNbO_3$ substrates.)

Effective waveguiding requires films with very low optical loss. Nominally, losses of less than 1 dB/cm are required. The sources of optical losses in thin films are; scattering by defects in the film, scattering by surface roughness, optical absorption and optical dispersion due to polycrystalline materials[23]. Therefore, low-defect density films are required, with low surface and interfacial roughness. The films must also have high purity and good oxygen stoichiometry (for low optical absorption) and must be single crystalline (for minimum optical dispersion). Another obstacle to implementing thin film $LiNbO_3$ devices is the lack of an efficient patterning technology. Unfortunately the high chemical stability of crystalline $LiNbO_3$ effectively precludes the use of standard photolithographic patterning techniques. Wet etching (using $HF/HNO_3$) is limited to several nm/min. Dry etching (RIE or RIBE) to 10's of nm/min. In fact, the present tool set available for processing bulk $LiNbO_3$ is limited to rudimentary functions (e.g. thermal diffusion) on bulk materials. As a consequence, it has not been possible to take advantage of the large refractive index and large optical nonlinearity of $LiNbO_3$. Demonstrated devices have thus far been based on weakly guiding waveguide structures.

### 1.3 UWM Two Stage Growth Process

UWM has developed a two-stage growth process for fabricating patterned structures of crystalline $LiNbO_3$ for photonic crystal and electro-optical waveguide devices[24]. The method uses MOCVD to deposit an amorphous $LiNbO_3$ film. The amorphous $LiNbO_3$ layer can be easily patterned using conventional photolithography and standard wet or dry etching techniques. For films deposited on crystalline $LiNbO_3$, the substrate serves as an effective etch stop. For amorphous films grown on $LiNbO_3$ substrates, a post deposition anneal converts the amorphous film to single crystal epitaxial $LiNbO_3$. We assume that similar epitaxial films would be obtained on closely lattice-matched substrates such as $LiTaO_3$.

As a preliminary feasibility experiment, UWM grew a 2 micron thick amorphous $LiNbO_3$ film on a z-cut $LiNbO_3$ substrate. The film was then patterned, with an orthorhombic 2D periodic pattern (7.6 x 10.1 $\mu m^2$), using standard photolithography and wet etching in dilute HF. Subsequent annealing at 1000 C resulted in the crystalline epitaxial $LiNbO_3$ structure shown in Figure 1.

Figure 2 is a cross-sectional TEM image of a similar amorphous $LiNbO_3$ film after annealing for 1 hour at 1100 C. The insert is the corresponding [01-10] zone axis selected area diffraction pattern, taken from the film/substrate interface area in the image, demonstrating the single crystal epitaxial nature of the layer. The inclined lines are bend contours; the horizontal band is a thickness fringe. **To our knowledge, this is the first time a 2 micron thick epitaxial $LiNbO_3$ film has been demonstrated.** The previous largest thickness reported was 0.6 microns for an epitaxial film on $LiTaO_3$[17]. These results demonstrate the capabilities of the UWM two stage process, namely the ability to make epitaxial $LiNbO_3$ films, thick enough for practical waveguide applications, and the ability to readily pattern these films to fine geometries.

**Figure 1:** Phase contrast Micrograph of an orthorhombic lattice (7.6 x 10.1 $\mu m^2$) patterned in epitaxial $LiNbO_3$.

Another major impediment to the implementation of $LiNbO_3$ thin films is the consistently low growth rates observed for films deposited by MOCVD or chemical beam epitaxy (CBE). Reasonable growth rates are an important consideration for commercial viability. In the course

the thin film work at UWM, the researcher identified what they believe is the source of the low deposition rates observed for the commonly used alkoxides precursors[25]. The culprit is an autocatalytic cycle involving hydrolysis and dehydration, which generates volatile monomers of Li and Nb, instead of stable oxides of the metal. Figure 3 shows the cycle for lithium butoxide. In the absence of this cycle, the estimated growth rates for LiNbO3 should be 5 to 10 times larger than the commonly observed; 0.2 μm/hr. Our recent studies of LiNbO3 MOCVD have confirmed these predictions, achieving growth rates up to 3.6 μm/hr. (See Part 4.1 of this proposal).

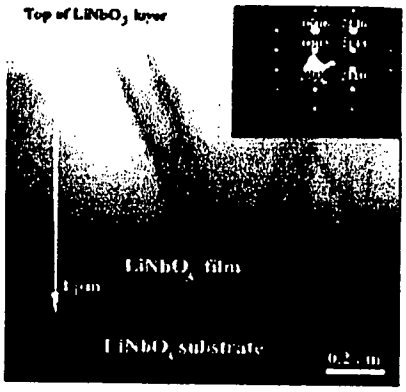


**Figure 2:** TEM of LiNbO3 film after annealing.

The UWM two stage process results in epitaxial $LiNbO_3$ films, of useful film thickness, and the ability to pattern these films to fine geometries. We have already demonstrated dramatically increases in the deposition rate for $LiNbO_3$, which will improve the commercial viability of the process. The other potential advantages of the process are those inherent to MOCVD. We can readily control the composition and composition profile of the films. High purity $LiNbO_3$ films should be achievable. (Bulk $LiNbO_3$ crystals frequently have Fe contamination). In addition, we are not limited to growth of the congruent ([Li]/[Nb]=0.94) composition, as in the case of the bulk material. An amorphous form of stoichiometric ([Li]/[Nb]=1.00) $LiNbO_3$ should be straightforward to deposit and anneal to crystallinity. Stoichiometric $LiNbO_3$ is known to have significantly smaller susceptibility to the photorefractive effect (i.e. an increase in the material's refractive index with exposure to visible light) and a five-fold lower voltage required for poling. We should also be able to obtain engineered doping profiles (e.g. with $Er^{3+}$) potentially leading to a totally new class of electro-optical devices.

### 1.4 Integrated Photonic Crystal Device - Electro-optically Gated Optical Add/Drop Multiplexer

As a first example of integration in nonlinear photonic bandgap (NLPBG) materials, we intend to fabricate a switchable 4-wavelength optical add/drop multiplexer (OADM). The optical circuit will be fabricated using 2D defect waveguides in a photonic crystal (~ 450 nm periodicity). These guides have the advantage of being able to provide very strong optical confinement. As a consequence, abrupt changes in direction of the light and very small radii of curvature, without loss of optical energy, are possible. This will permit a much higher degree of integration than is available with conventional index guides. A schematic of the proposed device is shown in Figure 3.

The degree of confinement is controlled by the number of lattice layers adjacent to the guiding region; evanescent coupling is produced by reducing the number of layers between guides. These waveguides have the added advantage of being able to be fabricated asymmetrically, i.e., with strong optical confinement on one side and weaker confinement on the other (to promote coupling). One of our tasks will be to determine the confinement per lattice layer ( ~ 1 dB/lattice layer in 3D photonic crystals – S. Fan, personal communication).

We are taking advantage of the electro-optic effect available in $LiNbO_3$ to perform the gating function. A microwave strip line and ground plane will be fabricated over the evanescent coupler

joining the feed waveguides and the resonators. Application of a voltage will change the relative propagation constant between the input/output waveguide and the resonator ring, decoupling the two. A second task of ours will be to determine the voltage-length product needed for decoupling.

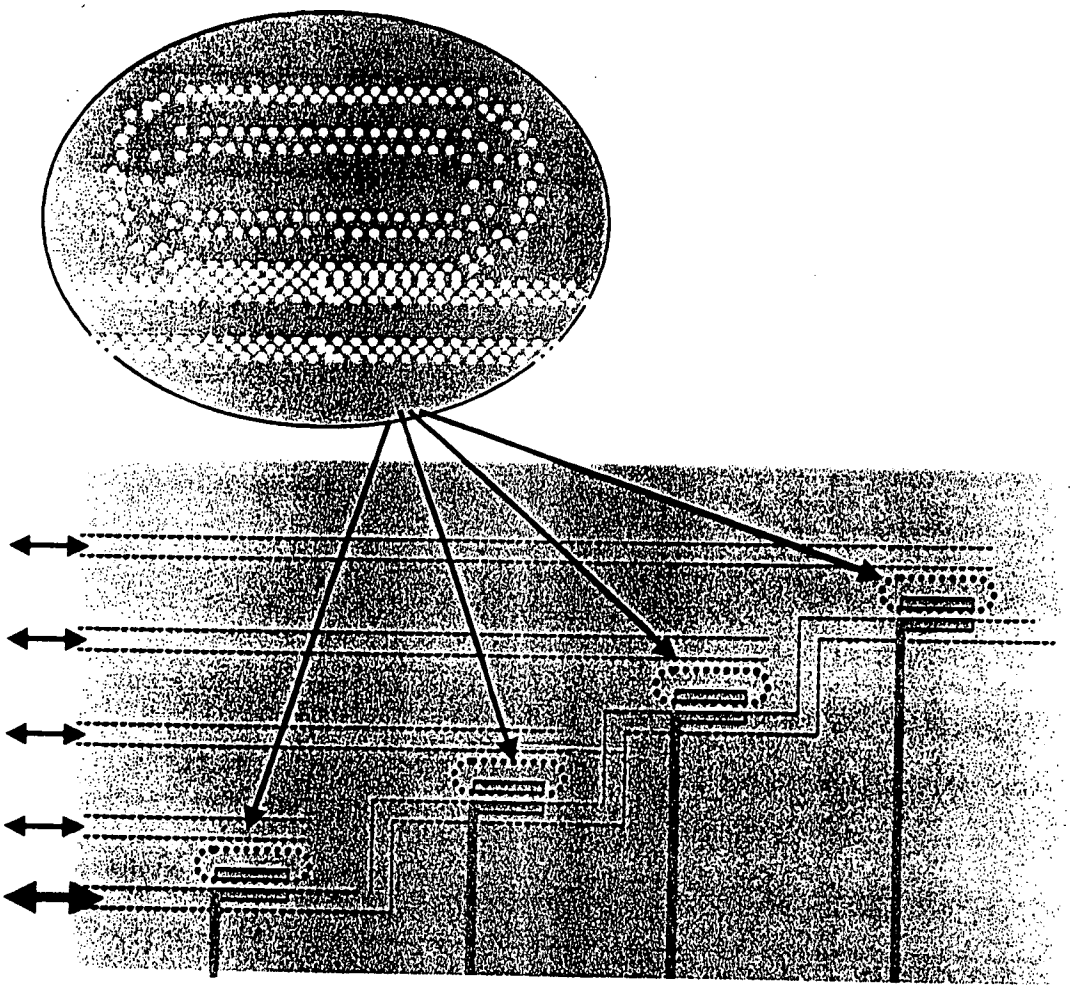

**Figure 3:** Schematic representation of the proposed 4-wavelength electro-optically gated optical add/drop multiplexer. The photonic crystal structures will be fabricated in epitaxial $LiNbO_3$ film. The microwave strip lines are vapor deposited and patterned gold.

If each resonator is to be responsive to only one wavelength on the ITU grid, then its free spectral range ($\nu_{FSR}=c/2nL$) need only be ~ 100 GHz. This corresponds to a maximum resonator path length of ~ 600 $\mu$m. Conventional photolithography of a much smaller length, say 60 $\mu$m, would provide sufficient center frequency accuracy. We also intend to investigate the use of the electro-optic effect to tune the resonator center frequency. The maximum resonator length to cover the C-band (1525 – 1565 nm) is about 15 $\mu$m. It should be possible to construct a defect waveguide resonator with this path length.

We estimate the device footprint for the integrated 4-channel add/drop multiplexer to be approximately 1 $mm^2$. If the same device were made using currently available technology (diffused waveguides in bulk $LiNbO_3$ crystals) device dimensions on the order of 10's of $cm^2$ would be required. The compact device is made possible only because of the excellent optical confinement possible with $LiNbO_3$ thin films, and our technology to deposit and pattern the $LiNbO_3$ photonic device structures.

## 1.5 Importance of the STTR Team

The investigators are uniquely suited to accomplish the goals of this program. McCaughan's group at UWM has been devoted to fabrication and characterization of $LiNbO_3$ based nonlinear optics and integrated optics for many years. This group was the first to demonstrate the use of nonlinear photonic crystals for telecommunications applications. Kuech, Saulys, and McCaughan have collaborated extensively in the areas of periodic poling mechanisms and $LiNbO_3$ film growth. Kuech is an internationally known expert both in CVD processes and reactor design. The elucidation of the film growth mechanism by Saulys and co-workers has led to the design and synthesis of more effective precursors and reactive carrier gases.

SMI is the leading US supplier of MOCVD systems for complex oxide thin films. SMI is currently developing fully integrated systems for Rotating Disc Reactor - Metal-Organic Chemical Vapor Deposition (RDR-MOCVD). We have used this technology to produce thin films and multilayers of a wide variety of complex oxide materials. Of particular interest to this effort is our related work on perovskite materials, such as $BaTiO_3$, $SrTiO_3$, $Ba_xSr_yTiO_2$ $SrBi_2Ta_2O_9$, $PbZr_xTi_{1-x}O_3$, and most notably $LiNbO_3$.

SMI and UWM have been teaming together for the past year, to develop and implement $LiNbO_3$ thin film technology. We have worked together on an MDA funded STTR effort to develop MOCVD hardware to deposit thick epitaxial $LiNbO_3$ films. We have also worked together on an Air Force funded STTR effort to develop photonic crystal devices utilizing the resulting $LiNbO_3$ films. (Please refer to Part 4.1 of this proposal for a detailed description of these efforts.) In this STTR effort, we propose to build on our previous accomplishments. We will use the process and hardware technology developed earlier to demonstrate integration of photonic devices into chip-scale optical network.

Pandanus Corporation was founded in 2001 to develop and commercialize the groundbreaking $LiNbO_3$ optical device technologies that originated at the University of Wisconsin. Pandanus original market studies indicated the potential for $500 million in component sales if fundamental $LiNbO_3$ device design limitations could be overcome. Pandanus is also building relationships with companies such as JDS Uniphase, to help with the insertion of these devices into today's fiber-optic telecommunication market. Pandanus Optical Technologies will license and commercialize the resulting photonic products developed under this effort.

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## *Part 2: Phase I Technical Objectives:*

| | **Phase I Objectives** |
|---|---|
| 1 | Demonstrate integration of photonic crystal device structures into chip scale optical networks, by building the electro-optically gated 4-channel add/drop multiplexer described herein. This work will utilize the epitaxial $LiNbO_3$ thin film technology developed earlier by the UWM/SMI team. |
| 2 | Identify critical issues for the chip scale integration technology for further development and resolution in Phase II. |

## *Part 3: Phase I Work Plan:*

| *Task Number* | *Task Description* | *Month* |
|---|---|---|
| 1 | Produce epitaxial $LiNbO_3$ thin film samples. | 1 - 4 |
| 2 | Characterize the $LiNbO_3$ samples for composition and crystallographic orientation. | 2 - 4 |
| 3 | Fabricate the chip-scale electro-optically gated 4-channel add/drop multiplexer. | 4 - 7 |
| 4 | Characterize the chip-scale electro-optically gated 4-channel add/drop multiplexer. | 7 - 8 |
| 5 | Design additional chip scale integrated devices for further development in Phase II. | 8 - 9 |
| 6 | Reporting. | 1 - 9 |

Task 1: Produce epitaxial $LiNbO_3$ thin film samples.

This task will be done at SMI. We will use the hardware developed earlier during our MDA funded STTR effort. We will also utilize the thin film deposition process technology, currently being developed under the Air Force funded SBIR effort. The UWM process initially deposits amorphous $LiNbO_3$ films. These films are then crystallized on subsequent annealing. Our standard process is to pattern the amorphous films prior to annealing, to take advantage of the higher etch rate of amorphous $LiNbO_3$.

For this task, the main challenge will be to produce epitaxial $LiNbO_3$ thin films. We have already demonstrated that homo-epitaxial $LiNbO_3$ films can be prepared on $LiNbO_3$ substrates. (Please see Part 1.3 of this proposal). For chip scale integrated optical networks, the ideal would be heteroepitaxial $LiNbO_3$ films on more practical substrates, such as sapphire or silicon. This will require the development of template layers. During this task, we will investigate template layers, deposited by MOCVD, for $LiNbO_3$ deposition on sapphire. We will anneal the as-deposited samples and characterize crystalline texture by x-ray diffraction. Our objective will be to define materials and processes to consistently produce heteroepitaxial $LiNbO_3$ thin films on sapphire substrates.

Our contingency plan will be to use a better lattice matched substrate, such as $LiTaO_3$. If we can not produce the desired heteroepitaxial films on any substrate, then we will use homoepitaxial films on $LiNbO_3$ substrates. We may still need to deposit a low index layer between the film and substrate to define the lower plane of the waveguide layer. Note that we could alternatively deposit doped $LiNbO_3$, for example with titanium, to also produce the waveguide films.

Task 2: Characterize the $LiNbO_3$ samples for composition and crystallographic orientation.

Characterization of the films will consist of x-ray diffraction to determine crystalline phase and texture, and chemical analysis to verify composition. X-ray diffraction can easily be done at either SMI or UWM. As we have found in our earlier work, accurate characterization of the Li/Nb ratio in the films is a challenge. Lithium is not an efficient emitter of x-rays. Thus techniques such as XRF and EDS do not work well. Also, SIMS is not straight forward, since lithium atoms tend to migrate in the film rather than sputter efficiently. In our current work, UWM is working to perfect ESCA techniques for analysis of $LiNbO_3$ films. If we can not identify an analytical technique that we have confidence in, the contingency plan will be to grow very thick $LiNbO_3$ films, remove and dissolve the film, and perform the analysis by atomic absorption. The characterization work will be done iteratively with Task 1. The overall objective is to produce films, with controlled composition, that consistently achieve epitaxy in the post-deposition annealed samples.

Task 3: Fabricate the chip-scale electro-optically gated 4-channel add/drop multiplexer.

This task will use the thin films developed in Task 1. The work will be done using The Wisconsin Center for Applied Microelectronics at UWM (See Part 8.1 of this proposal). The amorphous $LiNbO_3$ films will be patterned using optical photolithography and reactive ion etching. We will form the photonic crystal device structure, shown in Figure 3. The dimensions are well within the capabilities of the UWM facility. After patterning, the films will be annealed to produce crystalline epitaxial $LiNbO_3$ layers. We will then deposit and pattern gold films for the microwave strip lines, complete and package the device for testing.

Task 4: Characterize the chip-scale electro-optically gated 4-channel add/drop multiplexer.

Electro-optical characterization of the device will be done using the Integrated Optics Laboratory at UWM. (See Part 8.1 of this proposal). The objective will be to demonstrate efficient, high-speed signal multiplexing in the chip-scale integrated device.

Task 5: Design additional chip scale integrated devices for further development in Phase II.

On completion of Tasks 3 and 4, we will identify critical technical issues for chip scale integration of photonic devises using $LiNbO_3$ thin film. We will document these issues so that they can be investigated and resolved in Phase II. In addition, we will design additional chip scale networks, incorporating a variety of functional photonic crystal as well as electro-optical devices, for development in Phase II. The new networks will be designed to investigate and demonstrate resolution of the integration issues identified in Phase I.

Task 6: Reporting.

In accordance with the requirements of the Air Force, we will document all of our findings in interim reports and a final report. If invited, we will also present our plans for further development in a Phase II proposal.

Throughout this effort, both SMI and UWM will secure intellectual property rights for the hardware and processes by filing for patents, as appropriate. We will also disseminate technical information through technical publications, conference presentations, trade shows and through our product marketing efforts at SMI.

## *Part 4: Related Work:*

### 4.1 UWM/SMI Research on MOCVD of $LiNbO_3$ Thin Films

UWM and SMI have recently completed an MDA funded Phase I STTR effort titled "MOCVD System for $LiNbO_3$ Thin Film Waveguide Modulators and Optical Switches". We are also finishing an Air Force funded Phase I STTR effort titled "A Scaleable Method for Fabrication Nonlinear Photonic Crystals for Ultrafast All-optical and Electro-optic Functions". The currently proposed effort will build on the accomplishments of these projects. Figure 4.1.1 shows a photograph of the MOCVD test reactor at SMI, developed for $LiNbO_3$ deposition, in part using the MDA funds.

$LiNbO_3$ thin film growth studies were carried out at SMI, using a solution of precursors in toluene, injected into a flash evaporator. The precursor mix consisted of lithium *tert*-butoxide, purchased from Aldrich, and niobium ethoxide, purified by Prof. Saulys at UW. The metals ratio was Li/Nb =1.2/1. A total metals concentration of approximately 0.25 M was employed. The depositions were carried out at a substrate temperature of 500 C and a chamber pressure of 10

## INFORMATION ABOUT PRINCIPAL INVESTIGATORS/PROJECT DIRECTORS(PI/PD) and co-PRINCIPAL INVESTIGATORS/co-PROJECT DIRECTORS

Submit only ONE copy of this form **for each PI/PD and co-PI/PD** identified on the proposal. The form(s) should be attached to the original proposal as specified in GPG Section II.B. Submission of this information is voluntary and is not a precondition of award. This information will not be disclosed to external peer reviewers. ***DO NOT INCLUDE THIS FORM WITH ANY OF THE OTHER COPIES OF YOUR PROPOSAL AS THIS MAY COMPROMISE THE CONFIDENTIALITY OF THE INFORMATION.***

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**Gender:** ☒ Male ☐ Female

**Ethnicity:** (Choose one response) ☐ Hispanic or Latino ☒ Not Hispanic or Latino

**Race:** (Select one or more)
- ☐ American Indian or Alaska Native
- ☐ Asian
- ☐ Black or African American
- ☐ Native Hawaiian or Other Pacific Islander
- ☒ White

**Disability Status:** (Select one or more)
- ☐ Hearing Impairment
- ☐ Visual Impairment
- ☐ Mobility/Orthopedic Impairment
- ☐ Other
- ☒ None

**Citizenship:** (Choose one) ☒ U.S. Citizen ☐ Permanent Resident ☐ Other non-U.S. Citizen

**Check here if you do not wish to provide any or all of the above information (excluding PI/PD name):** ☐

**REQUIRED: Check here if you are currently serving (or have previously served) as a PI, co-PI or PD on any federally funded project** ☒

**Ethnicity Definition:**
**Hispanic or Latino.** A person of Mexican, Puerto Rican, Cuban, South or Central American, or other Spanish culture or origin, regardless of race.

**Race Definitions:**
**American Indian or Alaska Native.** A person having origins in any of the original peoples of North and South America (including Central America), and who maintains tribal affiliation or community attachment.
**Asian.** A person having origins in any of the original peoples of the Far East, Southeast Asia, or the Indian subcontinent including, for example, Cambodia, China, India, Japan, Korea, Malaysia, Pakistan, the Philippine Islands, Thailand, and Vietnam.
**Black or African American.** A person having origins in any of the black racial groups of Africa.
**Native Hawaiian or Other Pacific Islander.** A person having origins in any of the original peoples of Hawaii, Guam, Samoa, or other Pacific Islands.
**White.** A person having origins in any of the original peoples of Europe, the Middle East, or North Africa.

**WHY THIS INFORMATION IS BEING REQUESTED:**

The Federal Government has a continuing commitment to monitor the operation of its review and award processes to identify and address any inequities based on gender, race, ethnicity, or disability of its proposed PIs/PDs. To gather information needed for this important task, the proposer should submit a single copy of this form for each identified PI/PD with each proposal. Submission of the requested information is voluntary and will not affect the organization's eligibility for an award. However, information not submitted will seriously undermine the statistical validity, and therefore the usefulness, of information recieved from others. Any individual not wishing to submit some or all the information should check the box provided for this purpose. (The exceptions are the PI/PD name and the information about prior Federal support, the last question above.)

Collection of this information is authorized by the NSF Act of 1950, as amended, 42 U.S.C. 1861, et seq. Demographic data allows NSF to gauge whether our programs and other opportunities in science and technology are fairly reaching and benefiting everyone regardless of demographic category; to ensure that those in under-represented groups have the same knowledge of and access to programs and other research and educational oppurtunities; and to assess involvement of international investigators in work supported by NSF. The information may be disclosed to government contractors, experts, volunteers and researchers to complete assigned work; and to other government agencies in order to coordinate and assess programs. The information may be added to the Reviewer file and used to select potential candidates to serve as peer reviewers or advisory committee members. See Systems of Records, NSF-50, "Principal Investigator/Proposal File and Associated Records", 63 Federal Register 267 (January 5, 1998), and NSF-51, "Reviewer/Proposal File and Associated Records", 63 Federal Register 268 (January 5, 1998).

## List of Suggested Reviewers or Review rs Not To Includ (optional)

### SUGGESTED REVIEWERS:
Not Listed

### REVIEWERS NOT TO INCLUDE:
Prof. Bruce Wessels - Northwestern University

SMI also collaborates with Prof. Wessels on MOCVD of ferroelectric thin films. SMI has confidentiality agreements with both The University of Wisconsin and with Northwestern University. We have agreed to maintain the confidentiality of the University of Wisconsin results from Northwestern University and vice-versa.

# COVER SHEET FOR PROPOSAL TO THE NATIONAL SCIENCE FOUNDATION

PROGRAM ANNOUNCEMENT/SOLICITATION NO./CLOSING DATE/If not in response to a program announcement/solicitation enter NSF 03-2

NSF 02-056 01/22/03

FOR NSF USE ONLY

NSF PROPOSAL NUMBER

0320135

FOR CONSIDERATION BY NSF ORGANIZATION UNIT(S) (Indicate the most specific unit known, i.e. program, division, etc.)

DMI - SMALL BUS TECH TRANS PROGRAM

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223175106

SHOW PREVIOUS AWARD NO. IF THIS IS
☐ A RENEWAL
☐ AN ACCOMPLISHMENT-BASED RENEWAL

IS THIS PROPOSAL BEING SUBMITTED TO ANOTHER FEDERAL AGENCY? YES ☐ NO ☒ IF YES, LIST ACRONYM(S)

NAME OF ORGANIZATION TO WHICH AWARD SHOULD BE MADE

Structured Materials Industries, Inc.

AWARDEE ORGANIZATION CODE (IF KNOWN)

6200000249

ADDRESS OF AWARDEE ORGANIZATION, INCLUDING 9 DIGIT ZIP CODE

Structured Materials Industries, Inc.
201 Circle Drive
Piscataway, NJ. 08854

NAME OF PERFORMING ORGANIZATION, IF DIFFERENT FROM ABOVE

University of Wisconsin-Madison

PERFORMING ORGANIZATION CODE (IF KNOWN)

0038950000

ADDRESS OF PERFORMING ORGANIZATION, IF DIFFERENT, INCLUDING 9 DIGIT ZIP CODE

U of Wisconsin Madison
750 University Ave
Madison, WI 53706-1490

IS AWARDEE ORGANIZATION (Check All That Apply) (See GPG II.C For Definitions)
☒ SMALL BUSINESS ☐ MINORITY BUSINESS
☒ FOR-PROFIT ORGANIZATION ☐ WOMAN-OWNED BUSINESS

☐ IF THIS IS A PRELIMINARY PROPOSAL THEN CHECK HERE

TITLE OF PROPOSED PROJECT Low Voltage Ultrafast Traveling Wave Modulator

| REQUESTED AMOUNT | PROPOSED DURATION (1-60 MONTHS) | REQUESTED STARTING DATE | SHOW RELATED PRELIMINARY PROPOSAL NO. IF APPLICABLE |
|---|---|---|---|
| $ 100,000 | 6 months | 06/01/03 | |

CHECK APPROPRIATE BOX(ES) IF THIS PROPOSAL INCLUDES ANY OF THE ITEMS LISTED BELOW

☐ BEGINNING INVESTIGATOR (GPG I.A)
☐ DISCLOSURE OF LOBBYING ACTIVITIES (GPG II.C)
☒ PROPRIETARY & PRIVILEGED INFORMATION (GPG I.B, II.C.6)
☐ HISTORIC PLACES (GPG II.C.9)
☐ SMALL GRANT FOR EXPLOR. RESEARCH (SGER) (GPG II.C.11)
☐ VERTEBRATE ANIMALS (GPG II.C.11) IACUC App. Date _____
☐ HUMAN SUBJECTS (GPG II.C.11)
Exemption Subsection _____ or IRB App. Date _____
☐ INTERNATIONAL COOPERATIVE ACTIVITIES: COUNTRY/COUNTRIES INVOLVED (GPG II.C.9)
☐ HIGH RESOLUTION GRAPHICS/OTHER GRAPHICS WHERE EXACT COLOR REPRESENTATION IS REQUIRED FOR PROPER INTERPRETATION (GPG I.E.1)

PI/PD DEPARTMENT

PI/PD FAX NUMBER
732-885-5910

PI/PD POSTAL ADDRESS
201 Circle Drive
Suite 103
Piscataway, NJ 08854
United States

| NAMES (TYPED) | High Degree | Yr of Degree | Telephone Number | Electronic Mail Address |
|---|---|---|---|---|
| PI/PD NAME<br>Nick M Sbrockey | PhD | 1982 | 732-885-5909 | sbrockey@optonline.net |
| CO-PI/PD<br>Leon McCaughan | PH.D. | 1980 | 608-262-0311 | mccaughan@engr.wisc.edu |
| CO-PI/PD | | | | |
| CO-PI/PD | | | | |
| CO-PI/PD | | | | |



Electronic Signature

# CERTIFICATION PAGE

## Certification for Authorized Organizational Representative or Individual Applicant:

By signing and submitting this proposal, the individual applicant or the authorized official of the applicant institution is: (1) certifying that statements made herein are true and complete to the best of his/her knowledge; and (2) agreeing to accept the obligation to comply with NSF award terms and conditions if an award is made as a result of this application. Further, the applicant is hereby providing certifications regarding debarment and suspension, drug-free workplace, and lobbying activities (see below), as set forth in Grant Proposal Guide (GPG), NSF 03-2. Willful provision of false information in this application and its supporting documents or in reports required under an ensuing award is a criminal offense (U. S. Code, Title 18, Section 1001).

In addition, if the applicant institution employs more than fifty persons, the authorized official of the applicant institution is certifying that the institution has implemented a written and enforced conflict of interest policy that is consistent with the provisions of Grant Policy Manual Section 510; that to the best of his/her knowledge, all financial disclosures required by that conflict of interest policy have been made; and that all identified conflicts of interest will have been satisfactorily managed, reduced or eliminated prior to the institution's expenditure of any funds under the award, in accordance with the institution's conflict of interest policy. Conflicts which cannot be satisfactorily managed, reduced or eliminated must be disclosed to NSF.

### Drug Free Work Place Certification

By electronically signing the NSF Proposal Cover Sheet, the Authorized Organizational Representative or Individual Applicant is providing the Drug Free Work Place Certification contained in Appendix A of the Grant Proposal Guide.

### Debarment and Suspension Certification

(If answer "yes", please provide explanation.)

Is the organization or its principals presently debarred, suspended, proposed for debarment, declared ineligible, or voluntarily excluded from covered transactions by any Federal department or agency?

Yes ☐ No ☒

By electronically signing the NSF Proposal Cover Sheet, the Authorized Organizational Representative or Individual Applicant is providing the Debarment and Suspension Certification contained in Appendix B of the Grant Proposal Guide.

### Certification Regarding Lobbying

This certification is required for an award of a Federal contract, grant, or cooperative agreement exceeding $100,000 and for an award of a Federal loan or a commitment providing for the United States to insure or guarantee a loan exceeding $150,000.

### Certification for Contracts, Grants, Loans and Cooperative Agreements

The undersigned certifies, to the best of his or her knowledge and belief, that:

(1) No federal appropriated funds have been paid or will be paid, by or on behalf of the undersigned, to any person for influencing or attempting to influence an officer or employee of any agency, a Member of Congress, an officer or employee of Congress, or an employee of a Member of Congress in connection with the awarding of any federal contract, the making of any Federal grant, the making of any Federal loan, the entering into of any cooperative agreement, and the extension, continuation, renewal, amendment, or modification of any Federal contract, grant, loan, or cooperative agreement.

(2) If any funds other than Federal appropriated funds have been paid or will be paid to any person for influencing or attempting to influence an officer or employee of any agency, a Member of Congress, an officer or employee of Congress, or an employee of a Member of Congress in connection with this Federal contract, grant, loan, or cooperative agreement, the undersigned shall complete and submit Standard Form-LLL, "Disclosure of Lobbying Activities," in accordance with its instructions.

(3) The undersigned shall require that the language of this certification be included in the award documents for all subawards at all tiers including subcontracts, subgrants, and contracts under grants, loans, and cooperative agreements and that all subrecipients shall certify and disclose accordingly.

This certification is a material representation of fact upon which reliance was placed when this transaction was made or entered into. Submission of this certification is a prerequisite for making or entering into this transaction imposed by section 1352, Title 31, U.S. Code. Any person who fails to file the required certification shall be subject to a civil penalty of not less than $10,000 and not more than $100,000 for each such failure.

| AUTHORIZED ORGANIZATIONAL REPRESENTATIVE | | SIGNATURE | DATE |
|---|---|---|---|
| NAME<br>Gary S Tompa | | Electronic Signature | Jan 22 2003 4:29PM |
| TELEPHONE NUMBER<br>732-885-5909 | ELECTRONIC MAIL ADDRESS<br>gstompa@aol.com | | FAX NUMBER<br>732-885-5910 |

*SUBMISSION OF SOCIAL SECURITY NUMBERS IS VOLUNTARY AND WILL NOT AFFECT THE ORGANIZATION'S ELIGIBILITY FOR AN AWARD. HOWEVER, THEY ARE AN INTEGRAL PART OF THE INFORMATION SYSTEM AND ASSIST IN PROCESSING THE PROPOSAL. SSN SOLICITED UNDER NSF ACT OF 1950, AS AMENDED.

**NATIONAL SCIENCE FOUNDATION**
Program Solicitation/Instruction Guide Number **NSF 02-056**

## SBIR PHASE I - PROPOSAL COVER PAGE

| TOPIC | SUBTOPIC LETTER (If any) | TOPIC TITLE |
|---|---|---|
| **EL** | **C** | **Electronics** |

PROPOSAL TITLE **Low Voltage Ultrafast Traveling Wave Modulator**

| COMPANY NAME | EMPLOYER IDENTIFICATION NUMBER (EIN) OR TAXPAYER IDENTIFICATION NUMBER (TIN) |
|---|---|
| **Structured Materials Industries, Inc.** | **223175106** |

NAME OF ANY AFFILIATED COMPANIES (Parent, Subsidiary, Predecessor)

ADDRESS (Including address of Company Headquarters and zip code plus four digit extension)
**Structured Materials Industries, Inc.**
**201 Circle Drive**
**Piscataway, NJ. 08854**

| REQUESTED AMOUNT | PROPOSED DURATION | PERIOD OF PERFORMANCE |
|---|---|---|
| **$100000** | **6** | |

| **THE SMALL BUSINESS CERTIFIES THAT:** | Y/N |
|---|---|
| 1. It is a small business as defined in the solicitation. | Y |
| 2. It qualifies as a socially and economically disadvantaged business as defined in the solicitation. (FOR STATISTICAL PURPOSES ONLY.) | N |
| 3. It qualifies as a women-owned business as defined in the solicitation. (FOR STATISTICAL PURPOSES ONLY) | N |
| 4. NSF is the only Federal agency that has received this proposal (or overlapping or equivalent proposal) from the small business concern. If No, you must disclose overlapping or equivalent proposals and awards as required by this solicitation | Y |
| 5.SBIR: A minimum of two-thirds of the research will be performed by this firm in Phase I.<br>STTR: It will perform at least 40 percent of the work and the collaborating research institution will perform at least 30 percent of the work as described in the proposal. | Y |
| 6. The primary employment of the Principal Investigator will be with this firm at the time of the award and during the conduct of the research. | Y |
| 7. It will permit the government to disclose the title and technical abstact page, plus the name, address and telephone number of a corporate official if the proposal does not result in an award to parties that may be interested in contacting the small business for further information or possible investment. | Y |
| 8. It will comply with the provisions of the Civil Rights Act of 1964 (P.L. 88-352) and the regulations pursuant thereto. | Y |
| 9. It has previously submitted proposals to NSF. | Y |
| 10. It previously submitted this proposal (which was declined) and significant modifications have been made as described in the solicitation. | N |
| 11. It has received Phase II awards from the Federal Government. If "yes" provide a company commercialization history in the supplementary documents module. | Y |

**PRINCIPAL INVESTIGATOR / PROJECT DIRECTOR**

NAME **Nick M Sbrockey**

| SOCIAL SECURITY NO. | HIGHEST DEGREE / YEAR | E-MAIL ADDRESS |
|---|---|---|
| **not displayed intentionally** | **PhD/1982** | **sbrockey@optonline.net** |
| TELEPHONE NO. | FAX NO. | WEB ADDRESS |
| **732-885-5909** | **732-885-5910** | |

**COMPANY OFFICER (FOR BUSINESS AND FINANCIAL MATTERS)**

| NAME | TITLE | TELEPHONE NO. |
|---|---|---|
| **Gary S. Tompa** | **President** | **732-885-5909** |

**OTHER INFORMATION**

PRESIDENTS NAME **Gary S. Tompa** YEAR FIRM FOUNDED **1994**

NUMBER OF EMPLOYEES (Including Parent, Subsidiary, Predecessor)
AVERAGE PREVIOUS 12 MO.: **12** CURRENTLY: **12**

RESEARCH INSTITUTION NAME
**University of Wisconsin-Madison**

RESEARCH INVESTIGATOR NAME
**Leon McCaughan**

RESEARCH INVESTIGATOR TELEPHONE NO.
**608-262-0311**

PROPRIETARY NOTICE: See instructions concerning proprietary information.
**Check Here ☐ if proposal contains proprietary information.**

# Low Voltage Ultrafast Traveling Wave Modulator

**Abstract:**

This Small Business Technology Transfer Phase I project will demonstrate a low-voltage waveguide modulator device, capable of operation at speeds up to 40 Gb/s. Ultimately, this device will be capable of operating at speeds up to 100 Gb/s, with drive voltages as low as 4 volts. The enabling technology for these devices is a process for deposition and patterning of single crystal $LiNbO_3$ thin films, which was invented by our STTR partners at the University of Wisconsin. Previously, the full potential of $LiNbO_3$ electro-optical devices could not be realized, due to the limitations of producing them by diffusion processes in bulk crystals. The UWM technology opens the way for a new class of electro-optical devices.

Structured Materials Industries, Inc. (SMI) has a long history of developing MOCVD systems for complex oxide film growth. UWM will work with SMI to transition the epitaxial $LiNbO_3$ film technology to commercial viability. We will also partner with a commercial supplier of electro-optical components, to provide technical guidance during the Phase I/II efforts, and for eventual commercialization of the resulting products. Together, this team is well positioned to commercialize $LiNbO_3$ thin film waveguide devices.

**Statement Concerning NSF Review Criterion 2:**

Fiber optic networks are being implemented in industry, defense and domestic and international telecommunications. Our proposed technology will enable new products that will add increased speed, capacity and flexibility to growing optical communications networks. We anticipate the products developed from this effort to achieve a significant market share by the year 2005. In the longer term, this technology can also be applied to devices for all-optical computing systems, which also require single crystal films of non-linear materials such as $LiNbO_3$.

**Key Words:**

waveguides, waveguide modulators, OEIC, fiber optics, $LiNbO_3$, thin films, epitaxy, MOCVD.

**Topic Name and Subtopic:**

Electronics / El-C. Photonics, Opto/Magneto-electronic Devices and Systems

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For font size and page formatting specifications, see GPG section II.C.



*Proposers may select any numbering mechanism for the proposal. The entire proposal however, must be paginated. Complete both columns only if the proposal is numbered consecutively.

# Low Voltage Ultrafast Traveling Wave Modulator

**STTR Phase I proposal. Submitted t The National Science Foundation**
**In response to Topic:** EL-C: Photonics, Opto/Magneto-electronic Devices and Systems
January, 2003

## *Part 1: Identification and Significance of the Opportunity:*

### 1.1 Overview

To move beyond the current use of fiber optics as a point-to-point "telegraph" system will require the development of compact, high-speed, low-voltage waveguide modulators and optical switching devices. $LiNbO_3$ is a nearly ideal material for fabrication of these devices, due to its large refractive index, excellent transparency and excellent electro-optical properties. However, the full potential of this material has yet to be realized commercially. Practical $LiNbO_3$ device fabrication is thus far based on limited processing techniques applied to bulk material. The implementation of high-speed, low-voltage waveguide modulator devices has been severely limited by the absence of viable growth and patterning techniques for $LiNbO_3$ thin films.

Recently, our STTR partners at The University of Wisconsin - Madison (UWM) have developed a two-step process for producing waveguide structures in single-crystal, epitaxial $LiNbO_3$ thin films. The patented UWM process results in $LiNbO_3$ films of thickness and quality, suitable for electro-optical device applications. The UWM process also enables high deposition rates, and relatively simple sub-micron patterning, both of which impact commercial viability in a positive way. The resulting waveguide structures will serve as the basis for a new class of truly compact electro-optic devices, offering greater speeds and lower operating voltages.

In this effort, UWM and SMI propose to work together to implement and commercialize the UWM technology. UWM will perform the device design, fabrication and characterization. SMI will grow the $LiNbO_3$ thin film materials, and develop the commercial processing hardware. We will also work with potential end-users, including Pandanus Optical Technologies and JDS Uniphase, for the electro-optical device technology and ultimate device commercialization. Together, this team is well positioned to accomplish the objectives of this effort. In Phase I, we will demonstrate proof of concept for the low-voltage traveling wave modulator device based on epitaxial $LiNbO_3$ thin film. In Phase II, we will build and optimize these devices, as well as optimize the commercial hardware for their production. In subsequent activities, we will commercialize the resulting electro-optical devices and the requisite thin film processing systems.

### 1.2 Materials Issues for Electro-optical Devices

Modern communication is increasingly based on fiber optics. This is due to the fact that optical signals carry a higher information content (more bits/second) than conventional electrical signals[1,2]. Present day versions of electro-optical switches and modulators are based on bulk crystals of $LiNbO_3$. A third elemental species (typically titanium) is diffused into the crystal to define waveguide layers in the surface[3]. The concentration profile of these waveguide layers is

limited to the typical error-function shaped diffusion profile, and thus only graded index waveguides can be produced. As a consequence, the mode profiles of these devices are poorly optimized for electro-optic functions. In addition, the weak confinement effectively precludes densely integrated circuits (which would require serpentine structures of small radii of curvature). The end-result is devices that are large, slow, and require high operating voltages. Ideally, optical waveguide components would be fabricated from thin films of ferroelectric $LiNbO_3$. A thin film deposition technology, such as metal-organic chemical vapor deposition (MOCVD), would allow fabrication of step index waveguide structures, and permit the fabrication of structures with arbitrary dopant (e.g., Ti, Er3+, etc.) profiles. The result would be more-compact devices, with consequent lower loss, higher speeds, lower operating voltages, and a greater degree of device integration.

Previous attempts to make $LiNbO_3$ thin films have used sputtering[4,5], laser ablation[6-8], sol-gel processing[9], thermal plasma spray CVD[10,11], liquid phase epitaxy[12,13], chemical beam epitaxy[14] and MOCVD[15-22]. Although there have been many encouraging reports of epitaxial deposition, in general, the films suffer from being too thin and from having excessive optical losses. For effective waveguiding, the films thickness must be on the order of the communication wavelength (presently 1.55 microns). Epitaxial $LiNbO_3$ film deposition on sapphire has only achieved a thickness up to 2000 angstroms, due to cracking caused by the large thermal expansion mismatch with the substrate[17]. $LiTaO_3$ substrates have a much better thermal expansion match with $LiNbO_3$, but have only resulted in films up to 6000 angstroms thick[17]. Effective waveguiding also requires films with very low optical loss. Nominally, losses of less than 0.2 dB/cm are required. The sources of optical losses in $LiNbO_3$ thin films are impurities (e.g. $Fe^{2+} \rightarrow$ photorefractive effects), defects in the film, surface roughness, low oxygen stoichiometry, and crystalline inhomogeneities.[23] A chemical vapor deposition process can provide the required high purity, low-defect density films and low surface and interfacial roughness, as well as precise control over stoichiometry and doping profiles.

Another obstacle to implementing thin film $LiNbO_3$ devices is the lack of an efficient patterning technology. The high chemical stability of crystalline $LiNbO_3$ effectively precludes the use of standard photolithographic patterning techniques. As a consequence, no viable processes exist to take advantage of the large refractive index difference inherent in $LiNbO_3$/air interfaces. Demonstrated devices have thus far been based on weakly guiding waveguide structures. The UWM two-stage growth process (see below) allows high aspect ratio geometries to be produced for the first time in $LiNbO_3$. As a result, strongly confining waveguide structures (such as hairpin bends for serpentine integrated optic geometries and high-Q resonator rings) can now be fabricated in $LiNbO_3$. In addition, the index contrast ($LiNbO_3$:air = 2.2:1) is sufficiently large to permit 2D photonic bandgap devices to be constructed. The successful development of a technology for deposition and patterning of high quality $LiNbO_3$ thin films will enable a new class of truly compact electro-optic devices and circuits.

### 1.3 UWM Two Stage Growth Process

UWM has developed a two-stage growth process for fabricating patterned structures of crystalline $LiNbO_3$ for photonic crystal and electro-optical waveguide devices[24]. The method uses MOCVD to deposit an amorphous $LiNbO_3$ film. The amorphous $LiNbO_3$ layer can be easily patterned using conventional photolithography and standard wet or dry etching techniques. For films deposited on crystalline $LiNbO_3$, the substrate serves as an effective etch stop. For amorphous films grown on $LiNbO_3$ substrates, a post deposition anneal converts the amorphous

film to single crystal epitaxial $LiNbO_3$. We assume that similar epitaxial films would be obtained on closely lattice-matched substrates such as $LiTaO_3$.

In a recent experiment, researchers at UWM grew a 2 μm thick amorphous $LiNbO_3$ film on a z-cut $LiNbO_3$ substrate. The film was then patterned, with an orthorhombic 2D periodic pattern (7.6 x 10.1 $\mu m^2$), using standard photolithography and wet etching in dilute HF. Subsequent annealing at 1000 C resulted in the crystalline epitaxial $LiNbO_3$ structure shown in Figure 1.

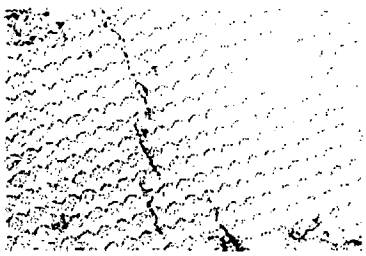


**Figure 1:** Phase contrast Micrograph of an orthorhombic lattice (7.6 x 10.1 $\mu m^2$) patterned in epitaxial $LiNbO_3$.

Figure 2 is a cross-sectional TEM image of a similar amorphous $LiNbO_3$ film after annealing for 1 hour at 1100 C. The insert is the corresponding [0110] zone axis selected area diffraction pattern, taken from the film/substrate interface area in the image, demonstrating the single crystal epitaxial nature of the layer. The inclined lines are bend contours; the horizontal band is a thickness fringe. **To our knowledge, this is the first time a 2 micron thick epitaxial $LiNbO_3$ film has been demonstrated.** The previous largest thickness reported was 0.6 microns for an epitaxial film on $LiTaO_3$[17]. These results demonstrate the capabilities of the UWM two stage process, namely the ability to make epitaxial $LiNbO_3$ films, thick enough for practical waveguide applications, and the ability to readily pattern these films to fine geometries.

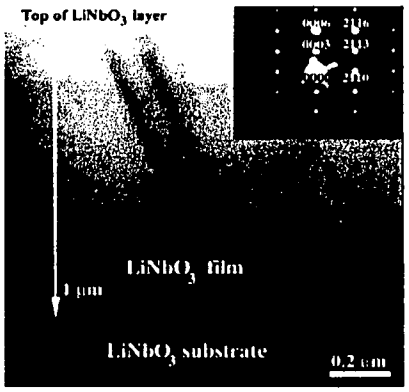



**Figure 2:** TEM of LiNbO3 film after annealing.

Another major impediment to the implementation of $LiNbO_3$ thin films is the consistently low growth rates observed for films deposited by MOCVD or chemical beam epitaxy (CBE). Reasonable growth rates are an important consideration for commercial viability. In the course of the thin film work at UWM, the researcher identified what they believe is the source of the low deposition rates observed for the commonly used alkoxides precursors[25]. The culprit is an autocatalytic cycle involving hydrolysis and dehydration, which generates volatile monomers of Li and Nb, instead of stable oxides of the metal. Figure 3 shows the cycle for lithium butoxide. In the absence of this cycle, the estimated growth rates for CBE would be some 5 to 10 times larger than the observed; ~ 0.2 um/h rate.

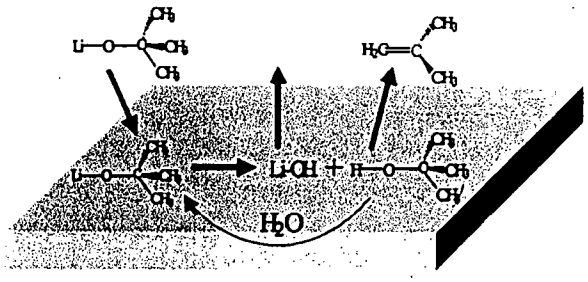



**Figure 3:** Model for the coupled hydrolysis and dehydration of lithium t-butoxide.

The key to overcoming this defect is to redesign the precursors such that they are either more stable in the presence of water, or decompose by a mechanism that removes the elements of water

from the surface before dehydration takes place. In principle, these next-generation precursors can be generated by either of two methodologies. A reactive carrier gas (e.g., $Me_3SiCl$) can be used to form more reactive precursor intermediates at the growing surface. Alternatively, and more directly, these precursor intermediates can be prepared in the laboratory and then introduced directly into the deposition chamber.

The UWM two stage process results in epitaxial $LiNbO_3$ films of useful film thickness, and the ability to pattern these films to fine geometries. We also believe we can dramatically increase the deposition rate for $LiNbO_3$, and improve the commercial viability of the process. The other potential advantages of the process are those inherent to MOCVD. We can readily control the composition and composition profile of the films. High purity $LiNbO_3$ films should be achievable. (Bulk $LiNbO_3$ crystals frequently have Fe contamination). In addition, we are not limited to growth of the congruent ([Li]/[Nb]=0.94) composition, as in the case of the bulk material. An amorphous form of stoichiometric ([Li]/[Nb]=1.00) $LiNbO_3$ should be straightforward to deposit and anneal to crystallinity. Stoichiometric $LiNbO_3$ is known to have significantly smaller susceptibility to the photorefractive effect (i.e. an increase in the material's refractive index with exposure to visible light) and a five-fold lower voltage required for poling. We should also be able to obtain engineered doping profiles (e.g. with $Er^{3+}$) potentially leading to a totally new class of electro-optical devices.

### 1.4 Device Applications

Current processing tools for $LiNbO_3$ devices are limited to thermal diffusion and electrode fabrication on bulk crystals. Our ability to grow, pattern, and re-crystallize $LiNbO_3$ thin films make it possible to produce a new generation of compact, highly integrated electro-optic devices, based on the ability to better confine both optical and microwave fields. (See Figure 4).

In this Phase I/II effort, we will focus on the development of an ultrafast, low-voltage, traveling wave modulator, as described below, since this device is anticipated to have the most significant near term market potential. However, the technology developed in this effort will be equally applicable to a wide variety of potential electro-optical devices. In the near term, these include tunable filters (also described below) and optical switches. Longer range applications include photonic crystal devices for communications and optical computing applications.

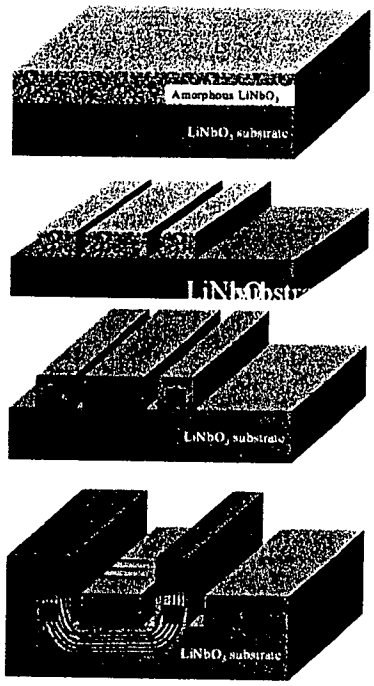



Figure 4. Schematic representing the deposition of α-$LiNbO_3$ lithography, etching, and annealing. The patterned trenches produce an improved field confinement and impedance.

<u>Low-Voltage, Ultrafast Traveling Wave Modulator</u>
Traveling wave modulators built from bulk $LiNbO_3$ crystals have been the most successful integrated electro-optic product to date. However, the inevitable demand for higher transmission bitrates (currently at 10 Gb/s), at reasonable drive voltages (under 4 volts), will require modifications to the device design beyond what can be accomplished in bulk. Our academic partner

(Leon McCaughan and UW-Madison) has carried out numerical simulations to explore thin film based devices for more near-term 40 Gb/s applications, as well as the feasibility of ultrafast modulators. As seen in the accompanying table, the patternable thin film process can be used to define trenches or wells (up to 11 μm deep) in the $LiNbO_3$ surface which improve the microwave-optical fields overlap by focusing the microwave radiation. These wells can also be used to modify the bulk dielectric properties of the $LiNbO_3$ at microwave frequencies. This latter aspect permits a better velocity matching between the two radiation fields. We note that we have recently developed the ability to fabricate very tall (in excess of 30 μm) electrode structures (via the photoresist SU-8). Calculations show that the standard gold versions of these electrodes would sufficiently reduce the microwave loss such that modulation bandwidths of up to 200 GHz can be achieved in a 4 cm long traveling wave modulator with only ~ 4 V of drive voltage. We are also exploring the use traveling wave electrodes made of copper, which is approximately 40% more conductive than gold (i.e., $g_{Cu}=5.8\times10^7\ [\Omega m]^{-1}$ vs. $g_{Au}=4.1\times10^7\ [\Omega m]^{-1}$). The UW lab is equipped with complete fabrication and characterization facilities, including optical and microwave test benches (with a 40 GHz network analyzer and optical spectrum analyzer). Several of the traveling wave modulator designs will be fabricated and optimized for a minimum of 47 GHz (40 Gb/s return-to-zero format).

Tunable High-Q Ring Resonators

Ridge structures provide the possibility of producing waveguides with large lateral confinement. This would permit the waveguide paths to be folded back on themselves, allowing both input and output fibers to be attached from a common end, thereby significantly reducing the device's footprint. The ability to fold waveguides would also allow long cascaded optical circuits to be folded back on themselves, reducing the device length.

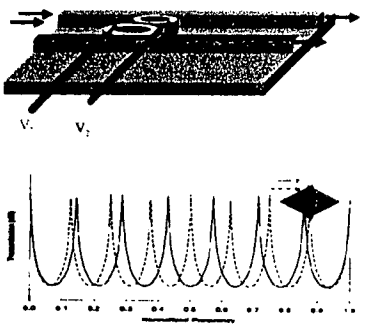
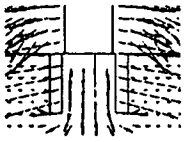


Figure 5. Electro-optically tunable filter via non-equivalent cascaded (vernier) rings.

Strong optical confinement also makes ring structures (single and cascaded) feasible, for applications such as WDM channel dropping filters, dispersion compensation, gain equalization, etc. Implementation of these structures in a nonlinear optic material such as $LiNbO_3$ would dramatically enhance the functionality of these devices: tunability by way of the electro-optic effect, frequency conversion and phase conjugation by way of 2nd order wave mixing. Figure 5 is a schematic of an electro-optically tunable filter using a pair of nonequivalent cascaded rings to provide a vernier action to the tuning[26].

Other applications of the patternable $LiNbO_3$ thin film technology can also be envisioned, including photorefractive-free thin films for visible integrated optic devices (such as RBG sources via second harmonic generation), and compact control of doping profiles during growth (e.g., for more efficient Er-doped waveguide amplifiers, vertical couplers, etc.

# Table I: Model Comparisons

| | Bandwidth | $V_{\pi(DC)}$ | $V_{3dBe}$ | Impedance |
|---|---|---|---|---|
| JDS bias-ready | 15 GHz | 5.5v | 5.6 v | 42Ω |
| Field-optimized | 15 | 2.6 | 3.8 | 46 |
| JDS | 30 (40Gb/s NRZ) | 6.1 | * | ? |
| Field-optimized | 30 | 2.8 | 4.0 | 48 |
| ---------- | -- | -- | -- | -- |
| Field-optimized | 47 (40Gb/s RZ) | 2.9 | 4.1 | 49 |

* X-cut, est. 9V, likely buffer layer for velocity match; no ridge.

## 1.5 Importance of the STTR Team

The investigators are uniquely suited to accomplish the goals of this program. McCaughan's group at UWM has been devoted to fabrication and characterization of $LiNbO_3$ based nonlinear optics and integrated optics. This group was the first to demonstrate the use of nonlinear photonic crystals for telecommunications applications. Kuech, Saulys, and McCaughan have collaborated extensively in the areas of periodic poling mechanisms and $LiNbO_3$ thin film growth. Kuech is an internationally known expert both in CVD processes and reactor design. The elucidation of the film growth mechanism by Saulys and co-workers has led to the design and synthesis of more effective precursors and reactive carrier gases.

SMI is the leading US supplier of MOCVD systems for complex oxide thin films. SMI is currently developing fully integrated systems for Rotating Disc Reactor - Metal-Organic Chemical Vapor Deposition (RDR-MOCVD). They have used this technology to produce thin films and multilayers of a wide variety of complex oxide materials. Of particular interest to this effort is our related work on perovskite materials, such as $BaTiO_3$, $SrTiO_3$, $Ba_xSr_yTiO_2$ $SrBi_2TaO_9$ and $PbZr_xTi_{1-x}O_3$.

Pandanus Corporation was founded in 2001 to develop and commercialize the groundbreaking $LiNbO_3$ optical device technologies that originated at the University of Wisconsin. Pandanus original market studies indicate the potential for $500 million in component sales if fundamental $LiNbO_3$ device design limitations could be overcome. Pandanus is also building relationships with companies such as JDS Uniphase, to help with the insertion of these devices into today's fiber-optic telecommunication market. Pandanus Optical Technologies will license and commercialize the resulting photonic products developed under this effort.

## *Part 2: Phase I Technical Objectives:*

The mission for the UWM / SMI team is to bring the UWM process technology to market. We will accomplish this in two steps. In Phase I, we will demonstrate technical feasibility. In Phase II, we will develop actual products. We will also partner with commercial organizations engaged in the development of fiber optic communications products. Pandanus Optical Technologies, of Madison, WI will provide technical input to the initial stages of this effort, and license and commercialize the resulting electro-optical devices during later program stages.

The UWM / SMI / Pandanus team is uniquely qualified to accomplish the goals of this program. Each brings specific capabilities to the effort. UWM provides the thin film process technology, and materials and device characterizations capabilities. SMI brings expertise in development and commercialization of MOCVD film deposition systems. Pandanus provides knowledge of optical communications technology and products. In addition, we will also partner with JDS Uniphase, who brings worldwide distribution capabilities to this effort.

The following Table summarizes the team's objectives for the Phase I program, as well as for Phase II and the eventual commercialization.

| | |
|---|---|
| **Phase I Objectives** | |
| 1 | Optimize the UWM two-step process using one of SMI's scaleable test MOCVD reactors, and produce $LiNbO_3$ thin films for subsequent characterization at UWM. The Phase I targets for the film deposition work are epitaxial $LiNbO_3$ films, at least 2 microns thick, deposited at rates of at least 1 micron/hour. |
| 2 | Demonstrate properties of the $LiNbO_3$ films that meet the requirements for compact, high-speed electro-optical devices. The Phase I targets are patterning and waveguide formation in the epitaxial $LiNbO_3$ films, demonstration of optical losses of 0.2 dB/cm or less, and fabrication and of the 40Gb/s low voltage (4 V or less) traveling wave modulator. |
| **Phase II Objectives** | |
| 1 | Develop and refine the $LiNbO_3$ MOCVD deposition process, to produce films suitable for electro-optic device fabrication, on wafer sizes up to six inches. The Phase II targets are epitaxial $LiNbO_3$ films, greater than 5 microns thick, deposited at rates of 5 micron/hour or better, with thickness uniformity better than 5% and optical losses less than 0.2 dB/cm. |
| 2 | Develop and demonstrate $LiNbO_3$ thin film low-voltage ultrafast waveguide modulator devices. The Phase II targets are data rates up to 200 Gb/s, at drive voltages of less than 4 volts. |
| **Commercialization Objectives** | |
| 1 | Commercialize $LiNbO_3$ thin film electro-optical devices through strategic partnerships with companies such as Pandanus Optical Technologies and JDS Uniphase. |
| 2 | Commercialize the MOCVD hardware to implement the UWM two step deposition process for fabrication of thin film $LiNbO_3$ devices. |

## *Part 3: Phase I Research Plan:*

| *Task Number* | *Task Description* | *Month* |
|---|---|---|
| 1 | Refine and demonstrate amorphous $LiNbO_3$ deposition using a scalable test MOCVD reactor at SMI. | 2 - 4 |
| 2 | Refine and demonstrate the annealing technology to produce epitaxial $LiNbO_3$ films from the amorphous deposits. | 3 - 5 |
| 3 | Fabricate waveguide structures in the resulting epitaxial LiNbO3 films. Characterize the films chemical, physical and optical properties and demonstrate their suitability for the low-voltage ultrafast waveguide modulator. | 4 - 5 |
| 4 | Deliverable - Phase I final report and Phase II proposal. | 6 |

Task 1: Refine and demonstrate amorphous $LiNbO_3$ deposition using a scalable test MOCVD reactor at SMI.

SMI will perform process optimization, starting with the UWM process and precursors. We will use low cost substrates (such as silicon or sapphire) for the initial process development. We will then use either $LiNbO_3$ or $LiTaO_3$ single crystal substrate, for demonstration of the optimized epitaxial film properties. The primary intent of this task will be to demonstrate a process to deposit amorphous $LiNbO_3$ films, at least two microns thick, at growth rates of at least one micron/hour. In the course of this activity, we will identify any hardware issues related to the scale-up of the process to commercial wafer sizes and production volumes. We will show proof of concept that we can resolve all scale-up issues. We will also investigate different $LiNbO_3$ film compositions, including both the congruent ([Li]/[Nb] = 0.94) and stoichiometric ([Li]/[Nb] = 1.00) film compositions. Bulk $LiNbO_3$ materials are limited to the congruent composition. However, the MOCVD process should allow us to deposit films of any composition. Selected films from this task will be provided to UWM for evaluation as described in Tasks 4 and 5 below.

Task 2: Refine and demonstrate the annealing technology to produce epitaxial $LiNbO_3$ films from the amorphous deposits.

UWM will refine and demonstrate the annealing technology to produce epitaxial $LiNbO_3$ films from the amorphous deposits produced at SMI in Task 1. Because $LiNbO_3$ is subject to $Li_2O$ out-diffusion during high temperature anneals. They have recently designed an annealing station which contains an overpressure of $Li_2O$. The goal of this task will be to demonstrate epitaxial films, of 2 micron thickness or greater, without stress related failures such as cracking. We will also demonstrate a process to pattern the amorphous deposits, and anneal them to produce pre-defined structures in the epitaxial $LiNbO_3$ films.

The resulting thin films will be characterized as needed at UWM. Chemical characterization will be made using SIMS. Structural characterization will be made principally via X-ray diffraction, with a limited number of TEMs to determine defect density. Maker Fringe analysis (a form of surface second harmonic generation) will be used to determine and monitor the Li/Nb ratio. These characterization results will be provided as feed-back to the on-going process development work at SMI.

Optical characterization will consist of fabricating channel waveguides and making propagation loss measurements, and determining the magnitude of the nonlinear optic coefficients via the electro-optic effect (e.g. using a standard Mach Zehnder waveguide interferometer). Our targets for this task are to obtain films with optical loss of 0.2 dB/cm or less, and electro-optic properties approaching that of bulk $LiNbO_3$. UWM is fully equipped to carry out all aspects of this characterization. See the Facilities Section of this proposal for a description of laboratory capabilities at UWM.

Task 3: Fabricate waveguide structures in the resulting epitaxial LiNbO3 films. Characterize the films chemical, physical and optical properties and demonstrate their suitability for the low-voltage ultrafast waveguide modulator.

Selected samples of the most promising epitaxial $LiNbO_3$ films will be fabricated into simple waveguide structures and devices at UWM. We will also provide samples to our partners at Pandanus Optical Technologies and JDS Uniphase (and other potential customers/partners that we identify) for their evaluation. The deliverable for this task will be verification that the films have the necessary properties for application in electro-optical devices, and a plan for the development of these devices in the Phase II effort.

Task 4: Reporting.

In accordance with the requirements of NSF, we will document all of our findings in a final report. If feasibility is demonstrated, we will also present our plans for further development in a Phase II proposal.

Throughout this effort, both SMI and UWM will secure intellectual property rights for the hardware and processes by filing for patents, as appropriate. We will also disseminate technical information through technical publications, conference presentations, trade shows and through our product marketing efforts at SMI.

## *Part 4: Commercialization Potential:*

*The product.* The focus of this effort will be low voltage ultrafast traveling wave modulators. However, the successful implementation of the UWM two-step process will also enable many other electro-optical devices, meeting both near term (40 Gb/s), as well as future (100 Gb/s and beyond) high speed communications demands. This technology could also enable eventual optical computing and all-optical communications products. The proposed technology will enable not only these devices, but a robust and production-worthy technology for their manufacture.

At the conclusion of the Phase II effort, we will commercialize the low voltage ultrafast modulator devices, and evaluate the other opportunities as appropriate. In addition to the devices, we also plan to sell high-volume manufacturing equipment for the epitaxial $LiNbO_3$ thin film processing, and non-exclusive licenses for the process technology.

*The Markets.* The world wide market for telecommunications components and equipment is anticipated to expand, after an initial decrease in 2001 and 2002. An approximate 35% annual growth rate is predicted over the next four years[27]. The market estimates reach $23 billion by 2005. In addition to the components market, there is also a market for the infrastructure (such as manufacturing equipment) to support this market. We plan to capitalize on these trends.

*The Competition.* The main competition to the proposed technology is from present day devices for optical telecommunications. These are primarily devices based on bulk $LiNbO_3$ crystals. The technology proposed in this effort will result in devices that are smaller, cheaper, operate at lower voltage and are capable of higher speeds. However, these technical advantages alone will not guarantee success, unless there is also a market pull for these products. Such market pull is difficult to see right now, due to the present overcapacity in the US telecommunications industry. However, the need for high speed optical communication devices still exists; particularly for specialized applications such as for military and homeland defense. Also, the demand for telecommunications products is still increasing. This market increase is real, although it is not readily apparent due to the present industry overcapacity. None-the-less, market demand will eventually catch up, most likely in the next 1 to 2 years. When the market does catch up, those companies with a technology advantage will be in a very good position.

MOCVD is the only thin film deposition technology that can potentially meet the needs of these products. SMI is the leading company specializing in MOCVD of complex oxide thin films. Our main competitors are Emcore, www.emcore.com, and Aixtron, www.aixtron.com, both of whom specialize in MOCVD systems for compound semiconductors. While both Emcore and Aixtron have competitive hardware technology, neither has significant process technology for thin film oxides. In addition, neither has significant expertise in end-uses involving piezoelectric or ferroelectric thin film materials.

*The Commercialization and Financing Plan.* A key element of our commercialization strategy is to first start with the right team. The team for this effort includes The University of Wisconsin at Madison, which has one of the foremost research efforts in the US for electro-optical thin films for device applications. UWM brings considerable expertise and existing process technology to this effort. Our team also includes Structured Materials Industries, Inc., who is the leading US company specializing in MOCVD systems for complex oxide thin film deposition. SMI brings expertise in the deposition hardware to this effort. Completing the team are Pandanus Optical Technologies and JDS Uniphase, who bring expertise in electro-optical devices and an existing market presence.

Our strategy will be to prove technical feasibility in Phase I, then develop the products in Phase II. In subsequent work, we will commercialize this technology at no cost to the government. We will first protect any intellectual property, by filling for the appropriate patents. Then, SMI will place a beta-system MOCVD reactor at Pandanus. Pandanus will use the SMI system and the UWM process technology to produces prototype traveling wave modulator products, and begin sampling these to customers. We will learn as much as we can from this development work and publicize the results as much as permissible. SMI and Pandanus will work collaboratively to disseminate the results of our efforts through (1) technical and trade publications, (2) advertising and presentations at trade shows, (3) presentations to potential customers and collaborators, (4) site visits to promising customers, (5) marketing through representatives and our respective websites and (6) through press releases. We will use this publicity to identify potential customers and other potential products that can be developed using the epitaxial $LiNbO_3$ thin film deposition and patterning technology.

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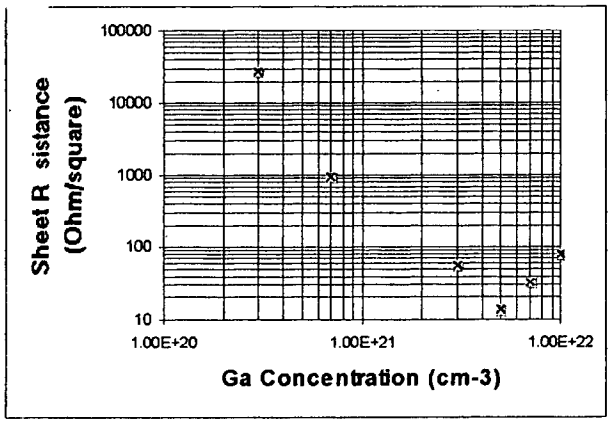
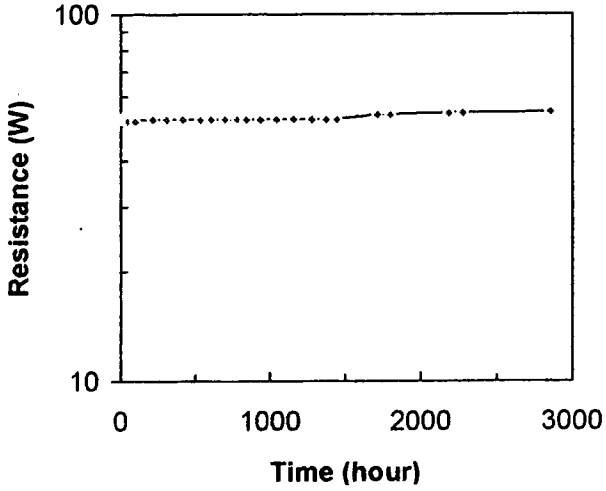
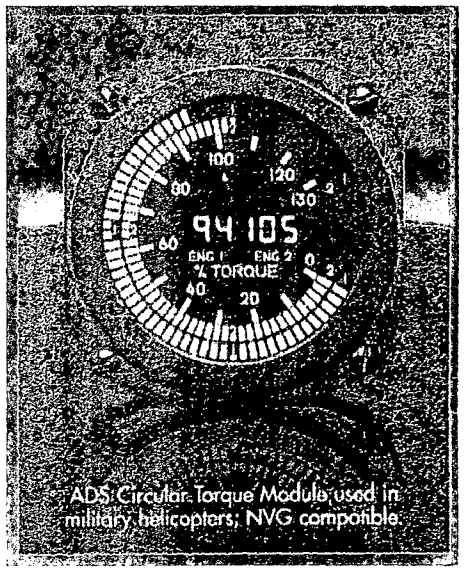
# *Focused Application TCOs*

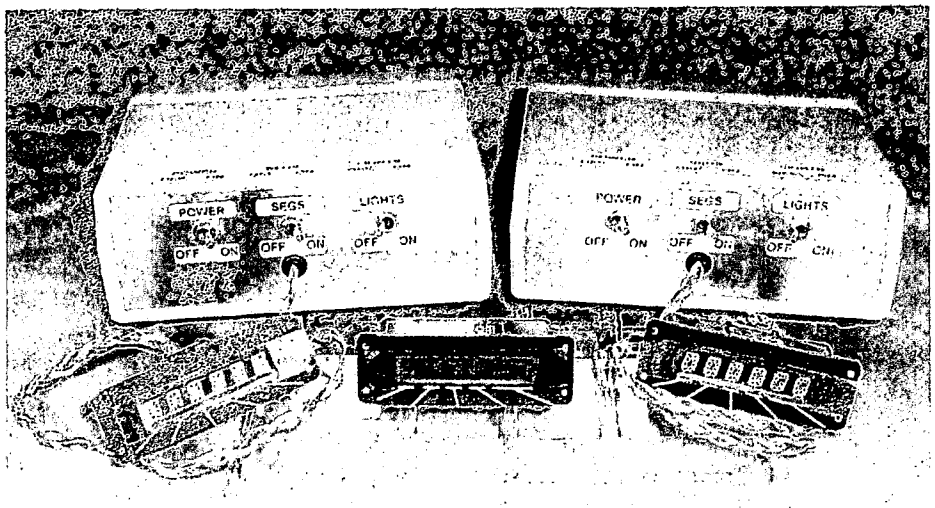
***Displays require Transparent Conductive Oxides (TCOs) for contact layers; heaters and insulators. (Oxides also have a strong potential as efficient phosphors)***

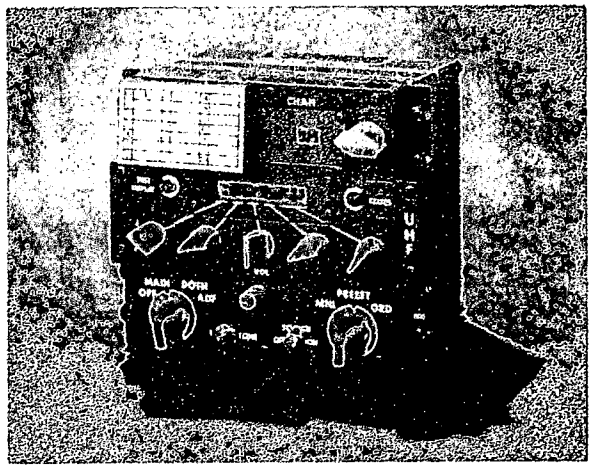


# Transparent Conducting Display Oxides

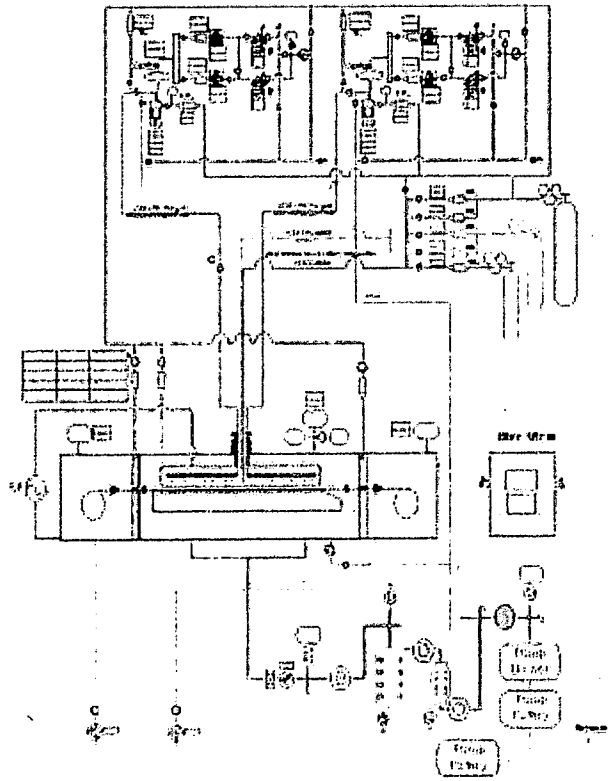


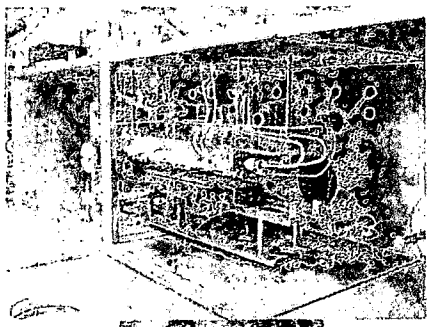
Advanced military and commercial displays require higher durability optical coatings

# MOCVD Tape Coating System

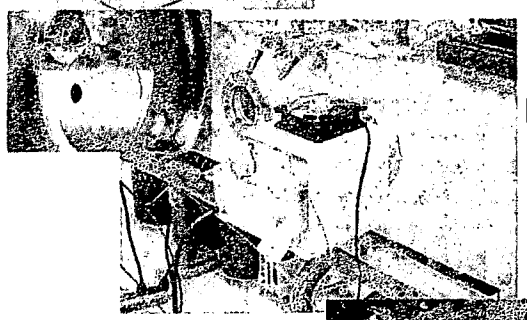
**Superconductors – Batteries – Circuit board components – an so on**



**MOCVD System Schematic**

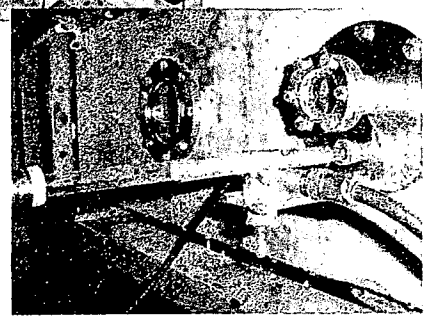


**MOCVD Assembly**



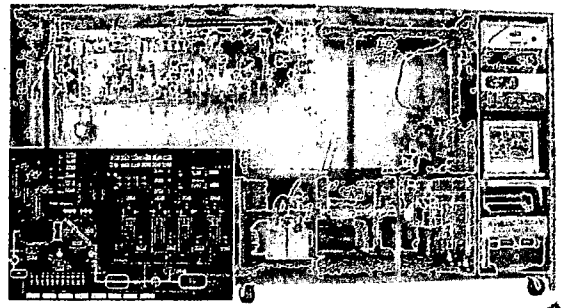
**UV Option**

**Plasma Option**



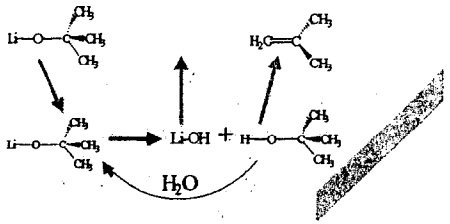
# *$LiNbO_3$ Photonic Crystal Devices*

Structured Materials Industries, Inc.

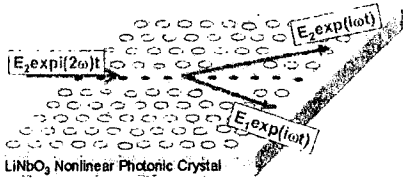


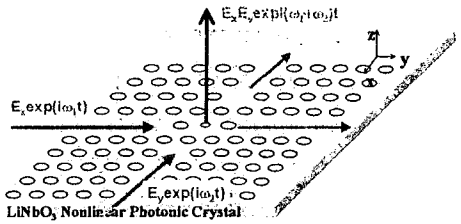
- Oxide MOCVD systems
- Wafer scaling

University of Wisconsin
& Corporate Sponsor




- Surface Chemistry
- PC design & fab.
- Device modeling



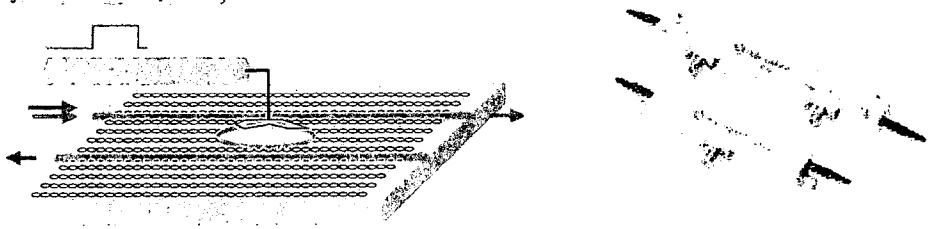




Phase contrast micrograph of an orthorhombic lattice (7.6 x 10.1 $\mu m^2$) patterned in $LiNbO_3$

## Targeted Customers

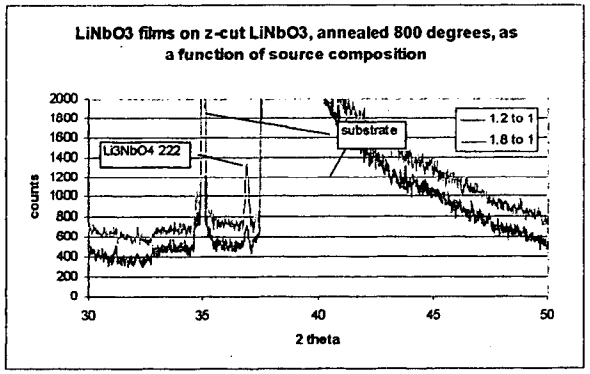
**JDS, Spectallis, ....**

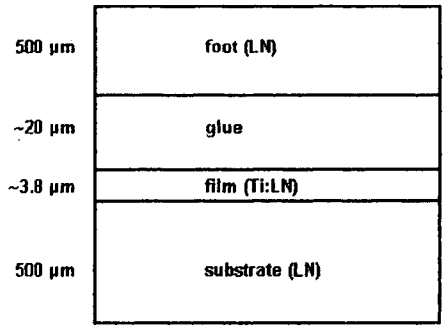
- Quantum communication
- Quantum computation
- Optical Data Manipulation
- Optical Logic

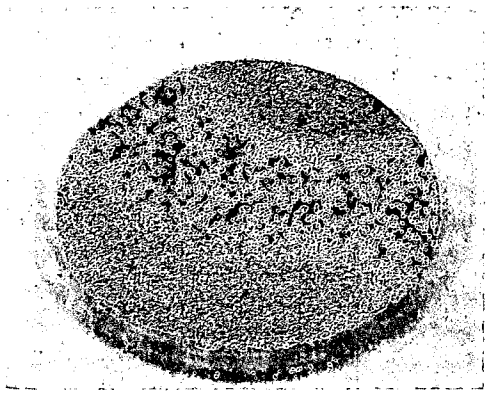


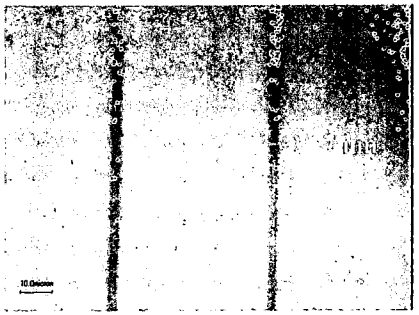
Gated Wavelength Filter

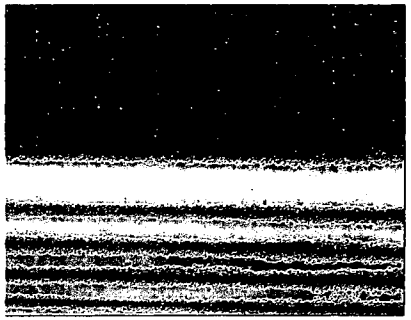
# $LiNbO_3$ properties



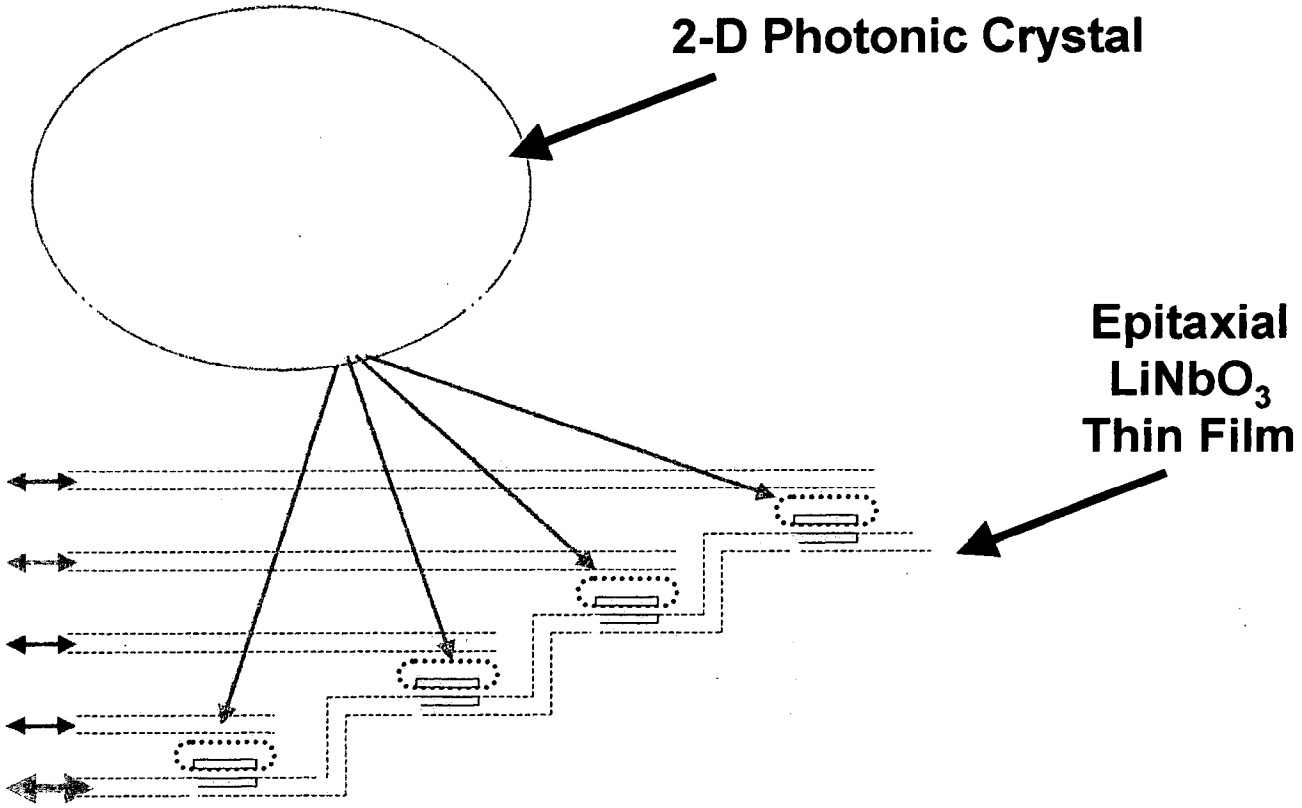







**Light guiding in Ti-doped $LiNbO_3$**

# Electro-Optically Gated Add/Drop Multiplexer



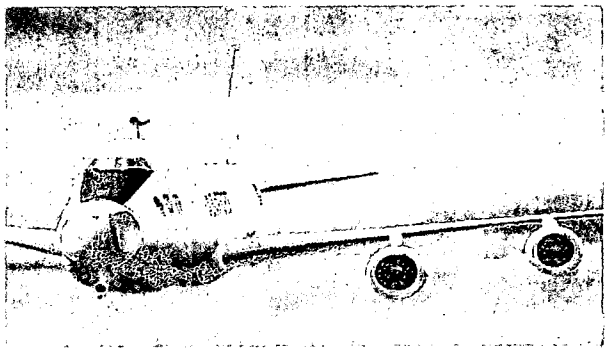
# *Very Low Absorption Coatings for High Power Laser Optics*

## Laser Damage Resistant Coatings for the Airborne Laser (ABL)

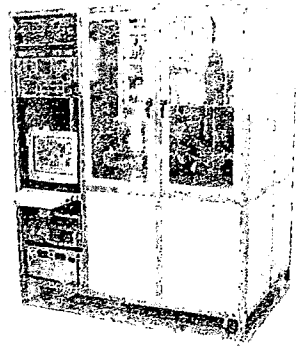
### Requirements

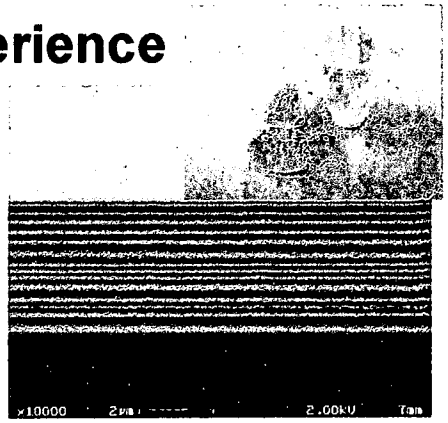
- Very Low Optical Absorption
- Low Residual Stress
- Refractory Material, Temperature and thermal Shock Resistant
- Phase I concepts proven, Phase II scale process

### Missile Defense System

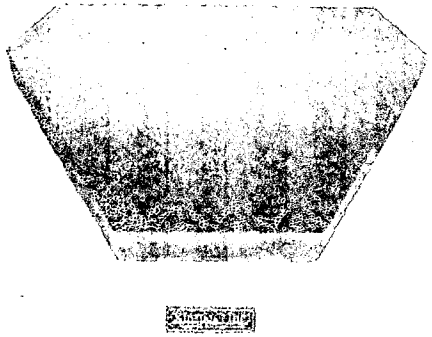


## SMI Reactor Capabilities & Coating experience






## RPE-MOCVD Coatings Properties



- High Purity
- Excellent Stoichiometry (Low Optical Absorption)
- Low Residual Stress
- Wide Range of Materials